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**Evaluation of Natural Pozzolans as Replacements for Class F Fly Ash in
Portland Cement Concrete**

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**Evaluation of Natural Pozzolans as Replacements for Class F Fly Ash in
Portland Cement Concrete**

by

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Dedication

I dedicate this to my parents, Maria and Tomas. Without your support and encouragement I could not have accomplished all I have so far.

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Abstract

Evaluation of Natural Pozzolans as Replacements for Class F Fly Ash in Portland Cement Concrete

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Most concrete produced today utilizes pozzolans or supplementary cementitious materials (SCMs) to promote better long term durability and resistance to deleterious chemical reactions. While other pozzolans and SCMs are available and provide many of the same benefits, Class F fly ash has become the industry standard for producing quality, durable concrete because of its low cost and wide-spread availability. With impending environmental and safety regulations threatening the availability and quality of Class F fly ash, it is becoming increasingly important to find viable alternatives. This research aims to find natural, lightly processed, alternatives to fly ash that perform similarly to Class F fly ash with regards to pozzolanic reactivity and provide comparable compressive strength, workability, drying shrinkage, thermal expansion properties and resistance to alkali-silica reaction, sulfate attack, and chloride ion penetration. Eight fly ash alternatives from the US were tested for compatibility with the governing standard for pozzolans used in portland cement concrete and various fresh and hardened mortar and concrete properties.

The results of this research indicate that six materials meet the requirements for natural pozzolans set by the American Society for Testing and Materials and many are comparable to Class F fly ash in durability tests. The primary concern when using these materials in concrete is the increase in water demand. The spherical particle shape of fly ash provides improved workability even at relatively low water-to-cement ratios; however, all of the materials tested for this research required grinding to achieve the appropriate particle size, resulting in an angular and rough surface area that requires more lubrication to achieve a workable consistency. So long as an appropriate water reducing admixture is used, six of the eight materials tested in this study are appropriate and beneficial for use in portland cement concrete.

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Chapter 1: Introduction

1.1 BACKGROUND

Concrete, a mixture of portland cement, coarse and fine aggregates, and water, is the most used man-made construction material in the world with nearly 3 tons used per person worldwide annually (Sustainability Benefits of Concrete, 2012). According to Mehta & Monteiro (2006), there are three primary reasons for the popularity of concrete as a construction material: 1) concrete can “withstand the action of water without serious degradation”, 2) concrete can be formed into an almost infinite variety of shapes and sizes, and 3) compared to other materials, concrete is readily available and relatively cheap. Part of the durability and cost benefits of concrete can be attributed to the use of supplementary cementitious materials (SCMs).

SCMs in use today are primarily waste products from other industries such as fly ash collected from coal burning power plants and ground-granulated blast furnace slag from steel production. These materials not only decrease the environmental impact of concrete by incorporating materials that would otherwise be thrown away but also improve the hardened properties of concrete made with them. Through the pozzolanic reaction, SCMs convert less desirable hydration products, like calcium hydroxide, to stronger, more durable products like calcium silicate hydrate (C-S-H) (Thomas, 2013); this improves the strength and decreases the porosity of the concrete made with SCMs. Although not all concrete produced worldwide incorporates SCMs, according to Thomas (2013), it is estimated that fly ash, the most widely used SCM in North America, is used in more than half of the all the concrete produced in the US. In fact, in a survey representing more than 75% of the coal consumed in 2011, the American Coal Ash Association (ACAA) found

that 13.8 million short tons¹ were used in concrete or blended cement (American Coal Ash Association, 2012). However, current and future US Environmental Protection Agency (EPA) restrictions on coal burning power plants have and will continue to impact the availability and usability of fly ash.

The EPA's 2005 Clean Air Interstate Rule (CAIR) and 2011 Cross-State Air Pollution Rule (CSAPR) both require the 27 states in the eastern half of the US to significantly reduce air pollution caused by coal burning power plants (US EPA, 2012; US EPA, 2013). Although the implementation of CSAPR is being held back by court hearings, CAIR is still in effect (US EPA, 2012). These two regulations focus on improving air quality in downwind states by reducing sulfur dioxide (SO₂) and nitrogen oxides (NO_x) emissions from coal burning power plants (US EPA, 2012; US EPA, 2013). Although there is no doubt that reducing these pollutants has improved the air quality in the US, the reduction techniques used by many power plants has made the fly ash generated of lesser quality if not unusable.

For example, a common solution for reducing SO₂ emissions is switching to a low SO₂ coal source. One such low SO₂ fuel source is known as Powder River Basin (PRB) coal sourced from Wyoming. While this coal does in fact reduce SO₂ emissions, the fly ash produced is characterized as high calcium (Class C) fly ash by ASTM C 618 (2012) (Tishmack, Olek, & Diamond, 1999). While Class C fly ash can be useful in certain circumstances, multiple studies have shown that Class C fly ash does not improve the resistance to various deleterious reactions in concrete in the same manner that Class F fly ash does (Thomas, 2013; Kruse, 2012; Jasso, 2012). Additionally, in addressing NO_x emissions, most power plants are required to retrofit their existing facilities with low NO_x

¹ 1 short ton = 2,000 lb

burners, which work by changing the combustion process. Because of these improvements, the resulting fly ash is not completely combusted and has significantly higher carbon contents than fly ash produced at the same facility with the same fuel source before low NO_x burners were installed (Hill, Sarkar, Rathbone, & Hower, 1997). The higher carbon content fly ashes are a problem for fly ash used in concrete because of the tendency for the remaining carbon to attract and retain air entraining admixtures used in concrete (Hill et al., 1997).

Furthermore, failure of the fly ash retaining pond in Kingston, Tennessee in December 2008 has generated concern regarding the health and safety hazards of fly ash. Because of this, the EPA is considering labeling fly ash as “special waste” classified by Subtitle C of the Resource Conservation and Recovery Act (US EPA, 2012). This would mean that fly ash would have cradle-to-grave regulation; everything from storage, to transportation, to disposal would be regulated, increasing the costs associated with using this material in concrete.

While the primary purpose of these enacted and proposed EPA regulations are beneficial to the health and safety of Americans, the practical implications on quality fly ash are detrimental to the durability and cost-benefit of concrete made with affected fly ash. Before the most stringent and cost-limiting regulations take effect, it is imperative that suitable pozzolanic alternatives be found that can be substituted for cement at percentages comparable to fly ash and produce acceptable concrete strengths and performance as good as, if not better, than concrete made with quality fly ash.

1.2 RESEARCH OBJECTIVE AND PLAN

This research aimed to identify natural pozzolans with potentially high availability that could be used as Class F fly ash alternatives. Materials marketed commercially as

natural pozzolans were tested along with materials known to have pozzolanic properties. All materials were characterized according to ASTM C 618 “Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete” (ASTM, 2012) to determine if they met the requirements for Class N pozzolans. Additionally, the fly ash alternatives were tested as SCMs in mortar and concrete to evaluate their ability to resist deleterious chemical reactions (alkali-silica reaction and sulfate expansion) as well as their effect on compressive strength, drying shrinkage, water demand, chloride penetrability, and coefficient of thermal expansion. An ASTM C 618 (ASTM, 2012) Class F fly ash was also tested in mortar and concrete to compare to the mortar and concrete made with the alternative materials.

1.3 THESIS STRUCTURE

This thesis is composed of four chapters, with this introduction as the first. Chapter 2 discusses all the materials used in this research, including the fly ash alternatives, and the results of the ASTM C 618 (2012) characterization. Chapter 3 presents the test methods, results, and discussion of the mortar and concrete testing. Results from previously published literature are included in the Results and Discussion sections of Chapter 3. Chapter 4 concludes this thesis with a summary of results, a decision on whether these materials are suitable for use in concrete, and recommendations for future studies.

Chapter 2: Materials and Characterization

2.1 MATERIALS

2.1.1 Cement and Fly Ash

The cement used for all paste, mortar, and concrete studies was an ASTM C 150 (ASTM, 2012) Type I cement produced by Texas Lehigh Cement Company in Buda, Texas. An ASTM C 618 (ASTM, 2012) class F fly ash from Rockdale, Texas was used as a supplementary cementitious material for comparison purposes in mortar and concrete studies. The chemical composition, measured by X-ray fluorescence (XRF), for both the cement and fly ash are shown in Table 2-1.²

Table 2-1: Chemical compositions of cement and fly ash

Oxide	Cement (wt %)	Fly Ash (wt %)
SiO ₂	19.1	52.1
Al ₂ O ₃	5.2	23.1
Fe ₂ O ₃	2.5	4.0
CaO	62.9	11.6
MgO	1.1	2.1
SO ₃	3.2	0.48
Na ₂ O	0.12	0.4
K ₂ O	0.91	0.74

2.1.2 Fine Aggregates

Three different fine aggregates were used during this research. For mortar and concrete mixtures, fine aggregates were primarily used as-received from the supplier except when the test method called for a specific gradation (ASTM C 1260/ASTM C 1567).

² Cliff Coward at TxDOT assisted with this work.

A standard graded sand (SFA) meeting the requirements of ASTM C 778 (ASTM, 2012) was used for most mortar testing including strength activity index, drying shrinkage, and expansion due to exposure to sulfate solution. As per ASTM C 778, this sand was sourced from Ottawa, IL. Absorption capacity and fineness modulus were not available for this material.

A known reactive fine aggregate (RFA) supplied by Wright Materials from Robstown, Texas was used for testing alkali silica reaction (ASR) resistance in mortars and concrete. This reactive fine aggregate was re-graded to meet the requirements of ASTM C 1260/ASTM C 1567 (ASTM, 2007; ASTM, 2013) for mortar testing and used as-received for concrete testing. Table 2-2 presents the absorption capacity, specific gravity, and fineness modulus and Figure 2-1 shows the as received gradation for the two fine aggregates used in the concrete mixtures.

A Colorado River sand supplied by Texas Industries from their Webberville quarry was the primary fine aggregate (FA) used for concrete testing. This fine aggregate was used as-received in the concrete mixtures.

Table 2-2: Aggregate properties

Aggregate	Absorption Capacity	Specific Gravity	Fineness Modulus
SFA	Not Available	2.65	Not Available
RFA	0.72%	2.58	2.14
FA	0.60%	2.62	2.73
CA	1.40%	2.65	--

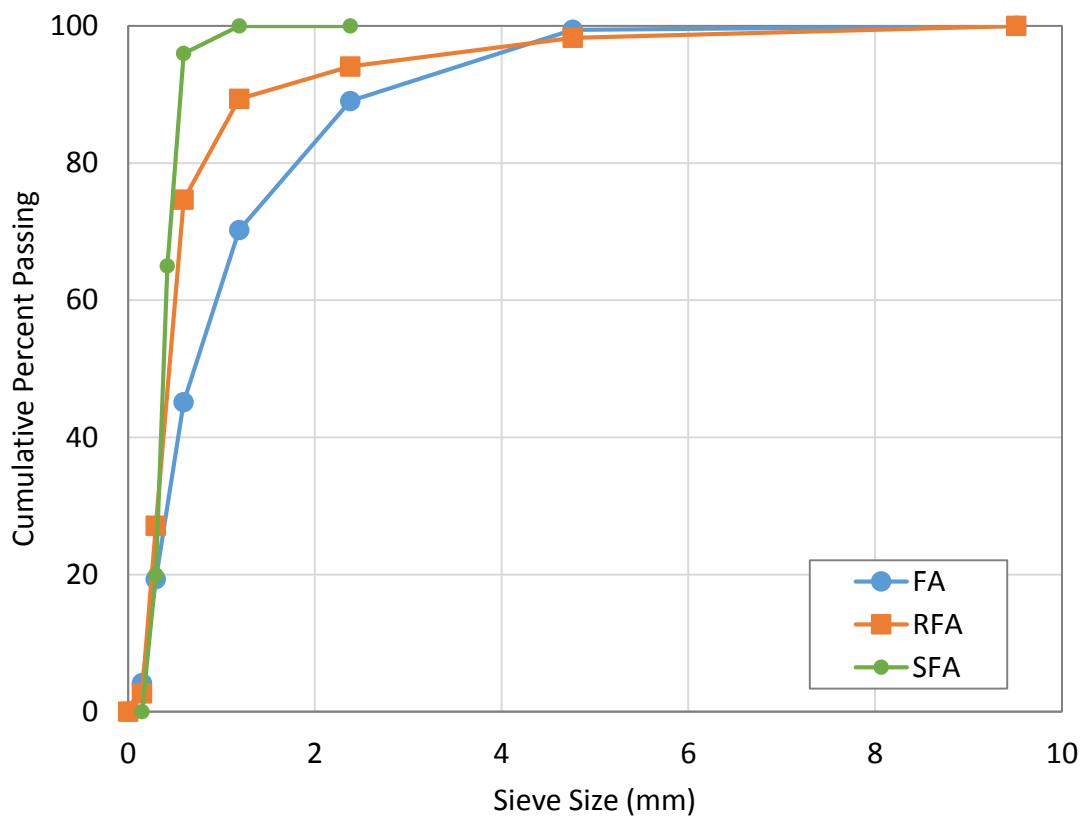


Figure 2-1: As-received gradation for FA, RFA, and SFA³

2.1.3 Coarse Aggregate (CA)

The coarse aggregate used for all concrete mixtures was a crushed, dolomitic limestone supplied by Texas Industries from their Bridgeport quarry. This aggregate was sieved and re-proportioned to meet the gradation requirements of TxDOT standard for coefficient of thermal expansion, Tex-428-A (TxDOT, 2011), and was used for all concrete mixtures including concrete made with reactive fine aggregate. The absorption capacity

³ SFA gradation shown based on ASTM C 778 (ASTM, 2012) requirements

and specific gravity for this coarse aggregate are included in Table 2-2. The gradation used for all concrete studies is shown in Figure 2-2.

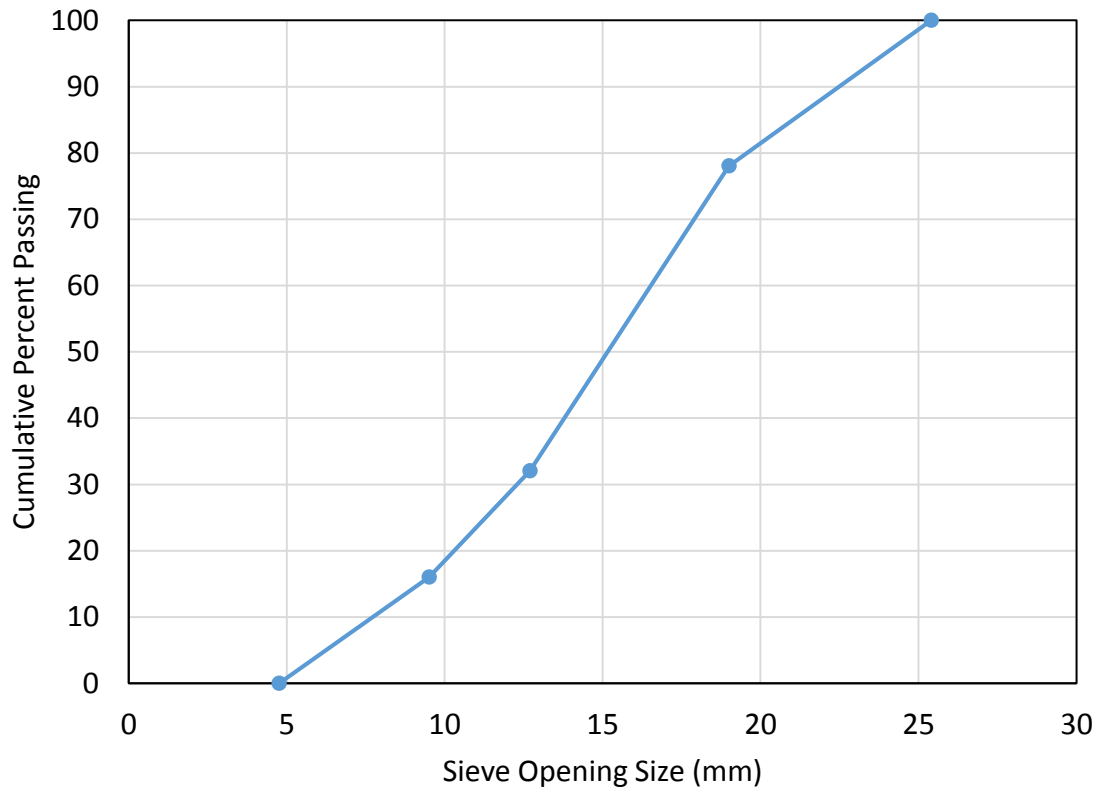


Figure 2-2: Coarse Aggregate Gradation

2.1.4 Admixtures

Two different water reducing admixtures (WRA) were used during this research. A polycarboxylate-based ASTM C 494 (ASTM, 2013) Type F WRA distributed by Sika Corporation under the trade name Sika ViscoCrete 2100 was used during preliminary screening in ASTM C 1260 (ASTM, 2007) mortar mixtures containing alternative SCMs. Some mixtures made with SCMs required more than the recommended dose of this WRA. For this reason, a different WRA was used in the concrete mixtures. The WRA used for

concrete studies was a naphthalene-based ASTM C 494 (ASTM, 2013) Type F WRA distributed by Sika Corporation under the trade name Sikament N.

2.1.5 Alternative SCMs

Eight SCMs were investigated as alternatives to Class F fly ash. Most of these materials were only quarried and ground; however, three materials were also calcined during processing; perlite, expanded shale, and metakaolin. Table 2-3 presents the supplier, trade name (when applicable), source, and mineral type for the SCMs studied. These materials were tested according to ASTM C 618 (ASTM, 2012) to determine if the as-received materials met criteria for Class N natural pozzolans and were also used in mortar and concrete to compare their performance to a Class F fly ash. Most materials were tested as-received; however, the expanded shale was received as a lightweight fine aggregate. This material was ground using a disc plate pulverizer to pass a No. 200 sieve before testing.

2.2 CHARACTERIZATION OF SCMs

ASTM C 618 (ASTM, 2012) is the governing specification for coal fly ash (Class C and F) and natural pozzolans (Class N) used in concrete. The criteria set forth in the ASTM specification are divided into three categories: 1) chemical requirements, 2) physical requirements, and 3) supplementary optional physical requirements. For the purposes of this study, uniformity criteria listed under physical requirements were not considered because the materials were collected and used from a single batch. Also, supplementary optional physical requirements were not considered for characterization, but some criteria will be discussed later under mortar studies. In addition to ASTM C 618 characterization,

the eight SCMs were tested for swelling clay content using a modified methylene blue test developed by W.R. Grace & Co.

Table 2-3: Alternative SCM supplier information

Material	Source	Mineral Type
Pumice	Idaho	Pumice
Perlite	Idaho	Perlite
Ash	Nevada	Vitric Ash
Metakaolin	Missouri	Metakaolin
Shale	Texas	Expanded Shale
Zeolite-1	Idaho	Zeolite
Zeolite-2	Texas	Zeolite
Zeolite-3	Texas	Zeolite

2.2.1 ASTM C 618 Chemical Requirements

2.2.1.1 Composition

ASTM C 618-12a (ASTM, 2012) requires a minimum combined silicon oxide, aluminum oxide, and iron oxide composition of 70.0% by mass and maximum sulfur trioxide composition of 4.0% by mass for Class N pozzolans. The specification states that these compositional requirements are meant only to describe the material and are not a measure of its reactivity. Fused pellets for XRF analysis were prepared in a Claisse M4 Fluxer according to TxDOT test procedure Tex-317-D (TxDOT, 2012) except that 0.5 g of SCM and 6.5 g of lithium borate-lithium bromide was used. The fused pellets were then analyzed in a Bruker S4 Explorer according to ASTM D 4326-11 (ASTM, 2011), as specified in ASTM C 311-11b (ASTM, 2011).⁴

⁴ Cliff Coward at TxDOT assisted with this work

2.2.1.2 Moisture content and Loss on Ignition (LOI)

Moisture content is the total weight lost upon drying at 110°C expressed as a percentage of the original weight and is conducted on as-received samples. LOI is the total weight lost when a dry sample is heated from 110°C to 750°C, expressed as a percentage of the moisture-free sample. ASTM C 618-12a (ASTM, 2012) specifies that Class N pozzolans must have a moisture content less than 3.0% by mass and an LOI less than 10.0% by mass. The moisture content of the eight materials was determined according to ASTM C 311-11b (ASTM, 2011), and LOI was determined according to the procedure described in ASTM C 114-11b (ASTM, 2011), as specified in ASTM C 311-11b (ASTM, 2011).

2.2.2 ASTM C 618 Physical Requirements

2.2.2.1 Fineness

Fineness is determined by wet-sieving an SCM through a No. 325 sieve and measuring the amount of material retained, expressed as a percentage of the original sample weight. ASTM C 618-12a (ASTM, 2012) requires that Class N pozzolans have less than 34% by mass retained on the No. 325 sieve after wet-sieving. As specified in ASTM C 311-11b (ASTM, 2011), this testing was conducted according the procedure described in ASTM C 430-08 (ASTM, 2008).

2.2.2.2 Strength Activity Index (SAI)

According to ASTM C 618-12a (ASTM, 2012), the strength activity index (SAI) is a measure of the reactivity of a given cement/SCM combination. SAI is measured by comparing the compressive strength of mortar cubes made with 20% cement by weight replaced with an SCM to a control mortar containing 100% cement. The compressive strength of the test specimen is expressed as a percentage of the control. ASTM C 618-12a requires an SAI of at least 75% measured at either 7 days or 28 days after mixing for Class

N pozzolans (ASTM, 2012). The mortars used for SAI testing were mixed according to ASTM C 305-11 (ASTM, 2012) and molded, cured, and tested according to ASTM C 109-11 (ASTM, 2012), as specified in ASTM C 311-11b (ASTM, 2011).

2.2.2.3 Water Requirement

Mortars containing SCMs mixed for SAI are required to have a water-to-cement ratio (w/c) such that the flow of the SCM mortar, measured according to ASTM C 1437 (ASTM, 2007), is ± 5 of the control mortar. ASTM C 618 specifies that the amount of water necessary to meet this flow requirement should not exceed 115% of the control (ASTM, 2012). Water requirement for each of the eight materials was determined according to ASTM C 311-11b (ASTM, 2011).

2.2.2.4 Soundness

The primary purpose of testing for soundness of a cement/SCM combination is to identify materials that have the potential to produce delayed expansion due to magnesium and calcium oxides. Soundness of a material is determined by measuring the autoclave expansion per ASTM C 151 (ASTM, 2009). In this method, specimens made of cement paste are exposed to high temperature and pressure for 3 hours after which the specimens are allowed to reach atmospheric pressure and are then cooled to room temperature. The expansion (or contraction) that occurs due to this process is expressed as a percentage of effective gage length. ASTM C 618 specifies that Class N pozzolans not have an autoclave expansion or contraction more than 0.8% (ASTM, 2012). Soundness testing was conducted on paste samples containing 20% SCM – 80% cement by weight. The pastes were mixed to normal consistency according to ASTM C 187-11 (ASTM, 2011) and tested according to ASTM C 151-09 (ASTM, 2009), as specified in ASTM C 311 (ASTM, 2011).

2.2.3 Modified Methylene Blue Testing

Aggregates contaminated with clays can cause workability problems in concrete mixtures because most natural clays have a tendency to absorb water. Because none of our materials are chemically-treated and only three are calcined, it was important to determine if these natural materials have a tendency to absorb water. A methylene blue test for swelling clay content in aggregates developed by W.R. Grace & Co. was modified so that absorption tendencies of the alternative SCMs could be evaluated. In this modified method, 20 g of standard graded sand (SFA) containing 5% by mass of an SCM is soaked in 30 g of a 5% by mass methylene blue solution for 5 minutes (1 minute agitation, 3 minutes rest, 1 minute agitation). After soaking, approximately 2 mL of the solution is transferred to a 3 mL syringe with a 0.2 μm luer-lok filter. The syringe is then depressed so that 0.5-1.0 mL of the solution is filtered into a new 1 mL vial. Using a micropipette, 130 μL of this filtered solution is then transferred to a new container where it is diluted with water to total weight of 45 g. This diluted solution is then mixed and transferred to a clean 16 mm glass tube. The methylene blue concentration in the diluted sample is then measured using a Hach DR 850 colorimeter. The output of the colorimeter is in units of mg methylene blue absorbed per g of sand. A control sample with 100% standard graded sand (SFA) was also tested to normalize the results.

According to ASTM C 33 (ASTM, 2013), fine aggregates are allowed to have up to 3% material by weight passing the No. 200 sieve. This standard also states that fine aggregates tested according to AASHTO standard T 330 (AASHTO, 2011) with methylene blue values of up to 5 mg/g are usually suitable for use in concrete. Although the test methods are not exactly the same, the underlying principle of both the AASHTO and modified Grace methylene blue tests are; each test will quantify the amount of methylene blue absorbed by the sample. Since the results of the two tests are essentially

interchangeable, the 5 mg/g methylene blue value was used for establishing a passing criteria for the fly ash alternatives. As an example, assume a concrete mixture has a fine aggregate content of 1400 lb/yd³ and a total cementitious material content of 550 lb/yd³. If no pozzolans were used, the concrete would have a total fines (passing the No. 200 sieve) content of 42 lb/yd³ with an allowable methylene blue content of 5 mg/g. If pozzolans were used at a 20% cement replacement dosage, the concrete would have a total fines content of 152 lb/yd³. This dramatic increase in fines content would result in a lower allowable methylene blue content of 1.4 mg/g. This was the maximum allowable methylene blue value for the specimens tested in this research. Although the author acknowledges the assumed mixture proportions are not suitable to describe all concrete mixtures, for the purposes of this study these proportions were deemed acceptable.

2.3 CHARACTERIZATION RESULTS AND DISCUSSION

2.3.1 ASTM C 618 Chemical Requirements

The results of the XRF analysis are shown in Table 2-4 along with moisture content and LOI results. These results show that all eight materials meet the compositional and LOI requirements of ASTM C 618 (ASTM, 2012). However, all three zeolites fail the ASTM C 618 moisture content requirement (ASTM, 2012).

2.3.2 ASTM C 618 Physical Requirements

Results for fineness, SAI, water requirement, and soundness are presented in Table 2-5; for comparison purposes, SAI and water requirement results for fly ash are also included in this table. These results show that pumice, perlite, metakaolin, ash, and shale meet all the physical requirements for ASTM C 618 Class N classification (ASTM, 2012). Zeolite-1 nearly meets all requirements, only missing the water requirement criteria by one

percentage point. Zeolite-2 and Zeolite-3 fail all ASTM C 618 physical requirements except soundness (ASTM, 2012).

2.3.3 Modified Methylene Blue Testing

The results of the modified methylene blue testing are shown in Figure 2-3. For comparison, a sample with 5% fly ash was also tested. The raw results were corrected for methylene blue absorbed by the standard graded sand (SFA) by subtracting 95% of the methylene blue value obtained from a 20 g sample of SFA; in other words, the results shown in Figure 2-3 represent the methylene blue absorbed only by the SCMs. Figure 2-4 shows the correlation between the ASTM C 618 water requirement and the results of the modified methylene blue testing. Based on the acceptance criteria described in Section 2.2.3, all fly ash alternatives except the three Zeolites have suitable methylene blue values.

Table 2-4: ASTM C 618 Chemical Analysis

Material	SiO₂ (wt %)	Al₂O₃ (wt %)	Fe₂O₃ (wt %)	Sum of Oxides (wt %)	CaO (wt %)	MgO (wt %)	SO₃ (wt %)	Na₂O (wt %)	K₂O (wt %)	Moisture Content (wt %)	LOI (wt %)
Pumice	69.4	12.4	1.1	82.9	0.94	0.44	0.04	3.8	5.2	1.5	4.4
Perlite	70.3	12.8	1.2	84.3	0.86	0.14	0.05	4.7	4.7	0.6	3.4
Ash	64.7	11.3	0.87	76.9	3.3	1.4	0.33	3.6	5.6	2.3	5.9
Metakaolin	51.7	35.2	2	88.9	0.57	0.45	0.06	0.1	1.4	0.9	1
Shale	65.4	14.6	5.7	85.7	2.4	2.3	0.39	1.1	2.9	0.3	0.4
Zeolite-1	65.3	10.9	2.4	78.6	2.5	0.59	0.07	0.52	4.8	5.1	2.5
Zeolite-2	59.5	12.9	2.2	74.6	5.1	0.82	0.29	3.1	2.6	4.8	4
Zeolite-3	62.2	11.9	1.1	75.2	2.2	0.64	0.14	1	1.7	11.6	4.6
ASTM C 618 Class N	--	--	--	> 70.0	--	--	< 4.0	--	--	< 3.0	< 10.0

Table 2-5: ASTM C 618 Physical Analysis

Material	Fineness % Retained	7 day SAI % Control	28 day SAI % Control	Water Req. % Control	Soundness % Expansion
Pumice	2	82	93	104	0.0
Perlite	2	86	94	100	0.0
Ash	15	72	83	102	0.0
Metakaolin	7	94	108	102	-0.1
Shale	30	72	81	103	-0.2
Zeolite-1	0	71	100	116	0.0
Zeolite-2	61	60	64	118	0.0
Zeolite-3	43	47	61	132	0.0
Fly Ash	--	79	87	93	--
ASTM C 618 Class N	< 34	> 75	> 75	< 115	< ±0.8

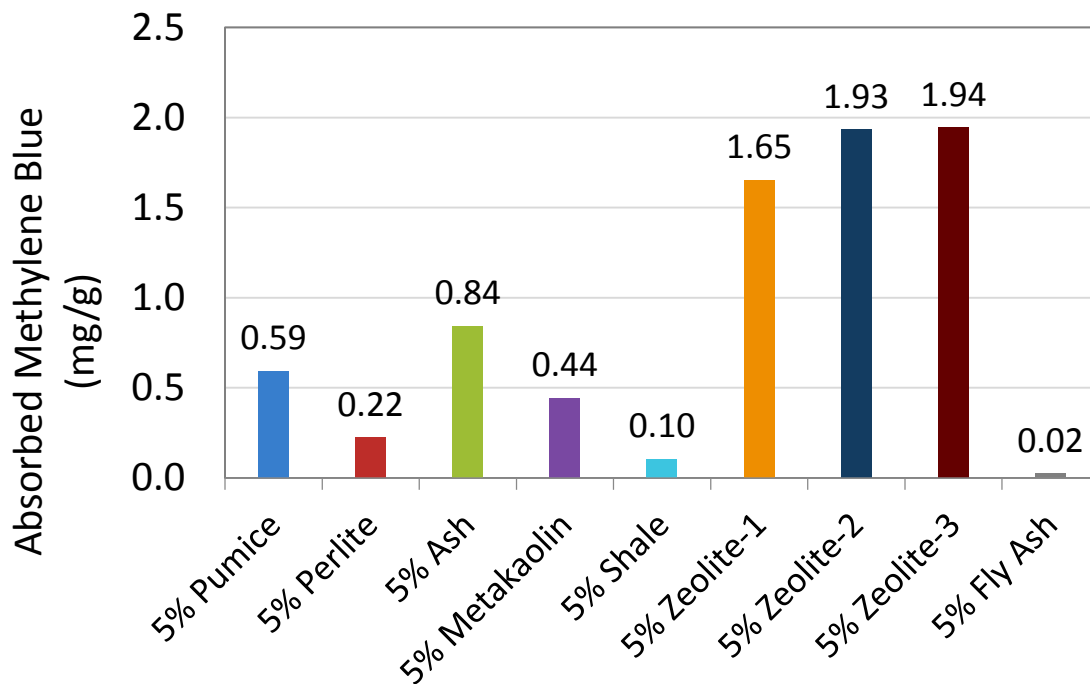


Figure 2-3: Modified methylene blue results corrected for sand absorption

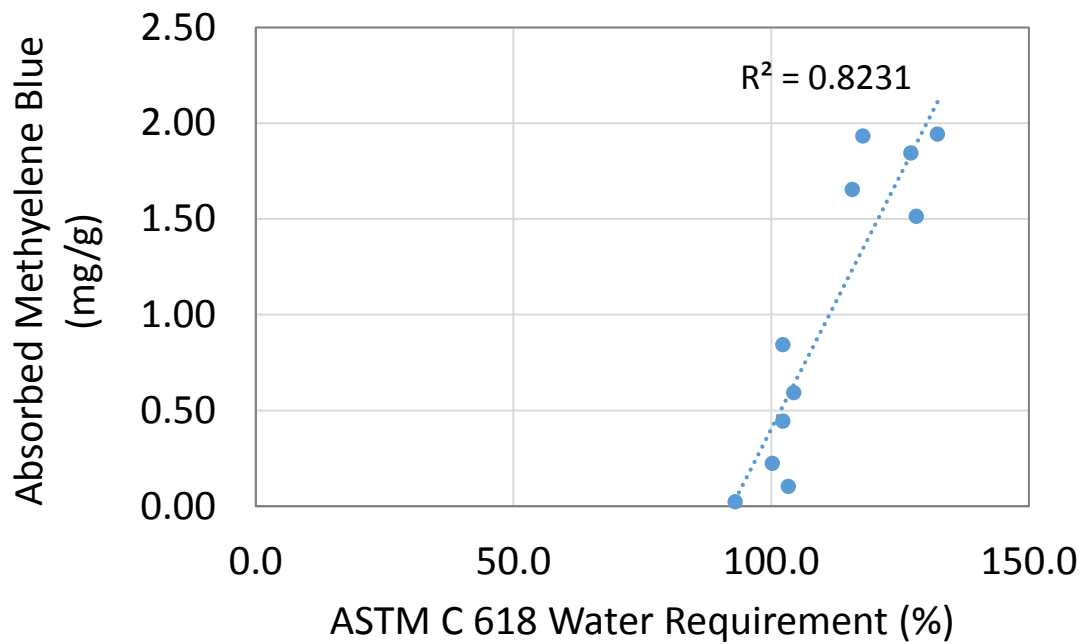


Figure 2-4: Relationship between modified methylene blue value and ASTM C 618 water requirement

2.3.4 Discussion of Natural Pozzolan Criteria

The results of the characterization tests show that pumice, perlite, metakaolin, ash, and shale meet all ASTM C 618 criteria for Class N pozzolans without any post-supplier chemical- or heat-treatment; by ASTM standards, these five materials are suitable for use in concrete. All three zeolites, however, failed ASTM C 618 moisture content and water requirement criteria; Zeolite-2 and Zeolite-3 also failed to meet fineness and SAI criteria. Zeolites in general are very porous and have a large, hydrophilic surface area (Snellings, Mertens, & Elsen, 2012 and Yilmaz, 2009). While these natural properties of zeolites may account for their high moisture content and water requirement, they do not explain the low reactivity shown in SAI for Zeolite-2 and -3. All characterization results are summarized in Table 2-6.

Table 2-6: Summary of Natural Pozzolan Results

Material	Sum of Oxides	Sulfur Trioxide	Moisture Content	LOI	Fineness	SAI	Water Requirement	Soundness	Methylene Blue
Pumice	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Perlite	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Ash	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Metakaolin	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Shale	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Zeolite-1	Pass	Pass	Fail	Pass	Pass	Pass	Fail	Pass	Fail
Zeolite-2	Pass	Pass	Fail	Pass	Fail	Fail	Fail	Pass	Fail
Zeolite-3	Pass	Pass	Fail	Pass	Fail	Fail	Fail	Pass	Fail

Chapter 3: Performance in Mortar and Concrete

After characterizing the eight SCMs based on the ASTM C 618 (ASTM, 2012) criteria for Class N pozzolans, it was important to determine if the performance of mortars and concrete made with these SCMs was comparable to the performance of mortars and concrete made with Class F fly ash. This chapter discusses test methods and results relating to ASR, compressive strength, workability, and drying shrinkage for mortars and concretes made with varying SCM content as a replacement for portland cement as well as expansion due to sodium sulfate exposure in mortars, rapid chloride penetrability (RCP) and coefficient of thermal expansion (CTE) for concretes containing fly ash alternatives.

3.1 ALKALI SILICA REACTION (ASR)

The high pH environment of concrete can cause the dissolution of reactive silica in some aggregates resulting in the formation of a hygroscopic, expansive gel. The formation of this silica gel can cause internal stresses that lead to reduced mechanical properties and durability of the affected concrete. Although there are treatments for concrete already showing signs of ASR, the easiest way to treat this problem is to attempt to prevent it entirely. Many studies have shown that Class F fly ash and other pozzolans are effective in reducing expansion caused by ASR when used in appropriate amounts (American Concrete Institute, 2012; Snellings, Mertens, & Elsen, 2012). This study first sought to determine the necessary cement replacement percentage to control ASR for each fly ash alternative using the accelerated mortar bar test (AMBT), ASTM C 1260/ASTM C 1567 (ASTM, 2007; ASTM, 2013). After determining the sufficient SCM content in mortars, concrete mixtures with similar SCM contents were tested according to ASTM C 1293 (ASTM, 2008) because ASTM C 1293 has been shown to more accurately predict ASR problems

in the field compared to the AMBT (Touma, Fowler, Carrasquillo, Folliard, & Nelson, 2001).

3.1.1 ASTM C 1260/ASTM C 1567 – Accelerated Mortar Bar Test

As the SAI results presented in Chapter 2 show, six of the eight materials had reactivities similar to fly ash. For this reason, the implications on strength gain were not considered the controlling factor when determining the optimum cement replacement percentage for each material; instead, a minimum cement replacement percentage for each material was determined by finding the SCM content necessary to limit expansion due to ASR of mortar mixtures to 0.10% after 14 days of 1 N sodium hydroxide (NaOH) exposure at 80°C, which is the threshold for controlling ASR stated in ASTM C1567 (ASTM, 2013).

To evaluate the ability of the different SCMs to control expansion due to ASR, 1 in. x 1 in. x 11 ¼ in. mortar bars with steel gage studs at each end were made according to ASTM C 1260 /ASTM C 1567 (ASTM, 2007; ASTM, 2013). As specified, a reactive sand, described in Section 2.1.2, was sorted, washed, and re-proportioned to the weight percentages shown in Table 3-1 and used as the fine aggregate. The standard requires a constant water-to-cementitious materials ratio (w/cm) of 0.47 and a measured flow of $\pm 7.5\%$ of a control mortar; a polycarboxylate-based superplasticizer, described in Section 2.1.4, was used to achieve the required flow. The admixture dosages for these mortar mixtures are shown in Table A-1 in Appendix A. After mixing and verification that the consistency was acceptable, the mortar was placed and compacted into the molds. After a 24 hour cure at 23 °C and 100% relative humidity, the mortar bars were removed from the molds, measured using a comparator (initial reading), submerged in water at room temperature, and placed in an oven set at 80 °C. After 24 hours in water at 80 °C the mortar bars were measured again (zero reading) and placed in a 1 N NaOH solution at 80 °C.

Additional readings were taken at 3, 7, 11, and 14 days after submersion in the NaOH solution. Expansion was calculated by determining the length change of the mortar bars expressed as a percentage of the gage length (10 in.).

Table 3-1: ASTM C 1260/ASTM C 1567 Grading Requirements
(ASTM, 2007; ASTM, 2013)

Sieve Size		Weight %
Passing	Retained On	
No. 4	No. 8	10
No. 8	No. 16	25
No. 16	No. 30	25
No. 30	No. 50	25
No. 50	No. 100	15

3.1.2 ASTM C 1293 – Concrete Prism Test

Concrete mixtures made with the minimum SCM content necessary to limit ASR expansion determined by the mortar testing described in Section 3.1.1 were mixed, cast, and tested according to ASTM C 192 (ASTM, 2012) and ASTM C 1293 (ASTM, 2008) except that the concrete mixture design was as shown in Table 3-2. The coarse and fine aggregates used for this testing were a dolomitic limestone and siliceous sand, described in Sections 2.1.3 and 2.1.2, respectively. All SCM contents are in weight percent; because all SCMs had specific gravities lower than cement, the volume of cementitious materials for all SCM mixtures was slightly greater compared to the control mixture. As specified by ASTM C 1293 (ASTM, 2008), NaOH was added to the concrete mixtures such that the alkali content of the concrete, expressed as Na_2O_e^5 , was 1.25% by mass of cement.

⁵ $\text{Na}_2\text{O}_e = (\text{wt \% Na}_2\text{O}) + 0.658 * (\text{wt \% K}_2\text{O})$

In this test, 3 in. x 3 in. x 11 ¼ in. concrete prisms with gage studs at each end were cast into molds and cured for 24 hours under wet burlap. At an age of 24 hours the prisms were de-molded, measured using a comparator, and placed vertically on elevated stands in felt-lined 5-gallon buckets filled with water to a depth of approximately 1 in. These containers were then placed in an environmental chamber set to 38 °C. At ages of 7, 28, and 56 days and 3, 6, 9, 12, 18, and 24 months the prisms were measured and their position within their respective containers was inverted so that the prisms were not stored with the same end up for two consecutive storage periods.

Table 3-2: Concrete Mixture Design for ASR Testing

Component	Batch Weight lb/yd ³	Weight %	Volume % ⁶
Coarse Aggregate	1937	48.3	43.4
Fine Aggregate	1257	31.3	28.9
Cementitious Material	564	14.1	10.6
Water	254	6.3	15.1
Air	--	--	2.0

3.1.3 Results and Discussion

3.1.3.1 Mortar

Initially, mortar mixtures were made with 20% cement by weight replaced with SCM. If the mortar for a given SCM performed well at 20% cement replacement by expanding less than 0.10% after 14 days of 1 N NaOH exposure, more mortars were mixed with decreasing SCM content until the measured expansion after 14 days was greater than 0.10%. If the mortar expanded more than 0.10% in mixtures with 20% SCM by weight of

⁶ These values are based on control concrete proportions. Mixture proportions for all ASTM C 1293 concretes are shown in Table B-1 of Appendix B.

cement, more mortars were made with increasing SCM content until the measured expansion after 14 days was less than 0.10%. The highest SCM content that yielded a 14 day expansion of less than 0.10% was considered the minimum necessary to mitigate ASR expansion.

Four materials, Pumice, Perlite, Metakaolin, and Zeolite-1, required 15% SCM by weight of cement to control expansion due to ASR. Three materials, Ash, Shale, Zeolite-3, required 25% SCM by weight of cement to control ASR expansion. Zeolite-2 required 35% cement replacement to minimize expansion to less than 0.10%. The results for all mixtures are summarized in Table 3-3; an “X” indicates that a particular SCM content was not tested while values in red indicate failure to meet the expansion criterion. Table 3-4 presents the minimum SCM content required to suppress expansion due to ASR and the percent reduction in expansion compared to a control mortar mixture with no cement replacement. Results for each material are shown in Appendix A.

The results of the initial mortar screening showed that, although some materials required a higher replacement dosage, all eight natural pozzolans were capable of controlling deleterious expansion due to ASR as measured by ASTM C 1260 /ASTM C 1567 (ASTM, 2007; ASTM, 2013). When compared to the ASTM C 311 (ASTM, 2011) fineness results reported in Chapter 2, it appears as though materials with less than 10% retained on a No. 325 sieve after wet sieving, Pumice, Perlite, Metakaolin, and Zeolite-1, performed better in the accelerated mortar bar test, requiring less than 20% cement replacement to meet the 0.10% expansion limit.

Table 3-3: Average Percent Expansion of ASTM C 1567 Mortar Bars after 14 Days
(X indicates that the combination was not tested; red values indicate that the combination failed the test)

Material	ASTM C 1260/ASTM C 1567 14 day Expansion, %					
	10% SCM	15% SCM	20% SCM	25% SCM	30% SCM	35% SCM
Pumice	0.16	0.04	0.00	X	X	X
Perlite	0.18	0.04	0.00	X	X	X
Ash	X	X	0.12	0.06	X	X
Metakaolin	0.23	0.06	0.02	X	X	X
Shale	X	X	0.11	0.07	X	X
Zeolite-1	0.20	0.02	0.01	X	X	X
Zeolite-2	X	X	0.25	0.26	0.14	0.07
Zeolite-3	X	X	0.16	0.09	X	X
Fly Ash	X	X	0.06	X	X	X

These mortar results generally agree with ASR mortar results in published literature. However, published results based on ASTM C 1260/ASTM C 1567 (ASTM, 2007; ASTM, 2013) or equivalent testing were not available for pumice or volcanic ash. The performance of Perlite in this study agreed with testing conducted by Bektas, Turanli, & Monteiro (2005); they found that finely ground perlite powder was effective in suppressing ASR expansion in mortars as measured by ASTM C 1260 (ASTM, 2007). Their study showed that as little as 16% natural perlite powder by weight of cement was necessary to limit ASR expansion to less than 0.10% after 14 days in NaOH solution when a “marginally reactive” aggregate was used (Bektas, Turanli, & Monteiro, 2005). Mortars made with Metakaolin in this research also agreed with results found in published literature. Ramlochan, Thomas, & Gruber (2000), using a Canadian standard similar to ASTM C 1260 (ASTM, 2007), found that mortar made with 15% cement replaced with metakaolin was effective in keeping ASR expansion below 0.10% after 14 days exposure to NaOH solution. Previous literature regarding the effect of zeolite on suppressing ASR

also supports the results of this research. Ahmadi & Shekarchi (2010) compared the ASR resistance of mortars containing natural zeolite to mortars containing a local fly ash (CaO 1.05% wt); their results showed that while 10% cement replacement with either natural zeolite or fly ash was not enough to limit expansion to 0.10% after 14 days NaOH exposure, 20% or more cement replacement with either SCM was effective in controlling ASR expansion. Finally, the results for mortar containing Shale in this study showed greater ability to control ASR expansion than calcined shale used in published reports. In a study using the National Building Research Institute (NBRI) Accelerated test for ASR, the basis for ASTM C 1260 (ASTM, 2007), Davies & Oberholster (1987) found that replacing 25% cement with calcined shale was not effective in controlling expansion; in fact, mortar bars with calcined shale behaved similarly to the control mortar bars.

Table 3-4: Results of ASR Mortar Testing

Material	Required SCM Content by Weight of Cement (%)	Reduction in Expansion From Control Mortar (%)
Pumice	15	90.5
Perlite	15	90.5
Ash	25	85.7
Metakaolin	15	85.7
Shale	25	83.3
Zeolite-1	15	95.2
Zeolite-2	35	83.3
Zeolite-3	25	78.6
Fly Ash	20	85.7

3.1.3.2 Concrete

Based on the results of the mortar testing, concrete mixtures containing 15%, 25%, and 35% SCM by weight of cement were made to evaluate the ability of each SCM to control expansion due to ASR. For materials that required only 15% cement replacement with SCM to control ASR expansion, a second concrete mixture containing 25% SCM by weight of cement was also tested. In addition, two concrete mixtures with 15% and 25% fly ash were made for comparison. At this time, only results up to 9 months are available; the specified testing period is 2 years. Figures 3-1 and 3-2 show the measured expansion for each concrete mixture at 28 days and 3, 6, and 9 months. Table 3-5 summarizes the 9 month results with respect to the control and fly ash concretes; concrete mixtures with 15% SCM content were compared to the 15% fly ash mixture while all others were compared to the 25% fly ash mixture. Full ASTM C 1293 (ASTM, 2008) results are available in Appendix B.

Although ASR testing in concrete will not be complete for more than a year, the preliminary results are encouraging. Concrete made with only 15% cement replaced with SCM reduced expansion by more than 80% compared to the control after 9 months of testing. Concrete made with higher SCM contents reduced expansion even more; most concretes with 25% or more cement replaced with SCM reduced expansion by more than 85% compared to the control after 9 months of testing. The 25% Ash and 35% Zeolite-2 concretes were the only two mixtures with greater than 25% cement replacement that reduced expansion by less than 85%. For the Pumice, Perlite, Metakaolin, and Zeolite-1 concretes, increasing the SCM content had varying effects. While there was a significant reduction in expansion when the SCM content was increased from 15% to 25% for the Pumice concretes, the concretes made with Perlite, Metakaolin, and Zeolite-1 at 25%

cement replacement had slightly higher 9 month expansions than at 15% replacements; the reason for this difference is not known, but the relationship may change over time.

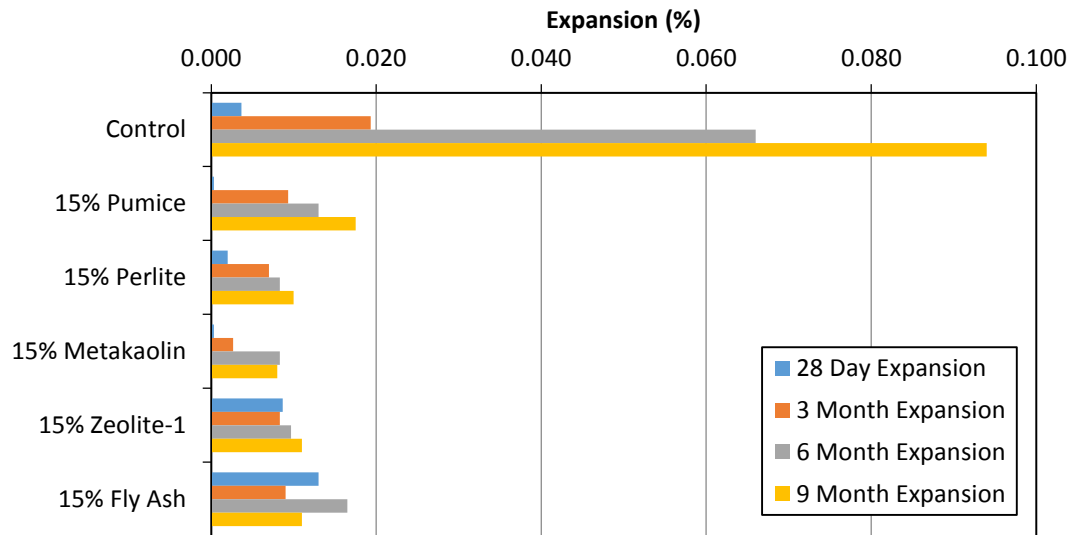


Figure 3-1: ASTM C 1293 Expansion for Low SCM Content

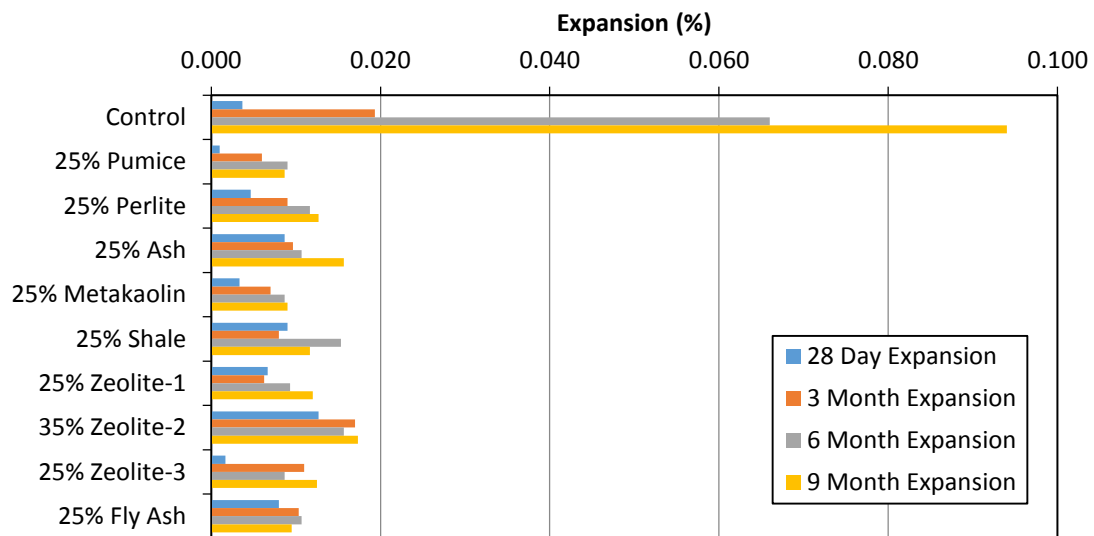


Figure 3-2: ASTM C 1293 Expansion for High SCM Content

Published data on the effects of pumice, perlite, ash, and zeolite on the ASR resistance of concrete were not available. However, results from published literature regarding the ASR performance of concretes containing metakaolin and shale support the preliminary results from this research. Ramlochan, Thomas, & Gruber (2000) used a Canadian standard similar to ASTM C 1293 (ASTM, 2008) to assess the ability of high-reactivity metakaolin (HRM) to control expansion due to ASR in concrete prisms; their results showed that concrete incorporating 15% or more HRM by weight of cement had less than 0.04% expansion after 2 years of testing. Additionally, field tests reported by Davies & Oberholster (1987) showed that that concrete prisms with 16.7 wt% calcined shale replacement of cement kept ASR expansion below 0.05% almost twice as long as the control.

Table 3-5: Summary of 9 Month Expansion Results

Concrete Description	9 Month Expansion, %	Percent Reduction from Control Concrete	Percent Reduction from Fly Ash Concrete
15% Pumice	0.018	81.4	-59.1
15% Perlite	0.010	89.4	9.1
15% Metakaolin	0.008	91.5	27.3
15% Zeolite-1	0.011	88.3	0.0
15% Fly Ash	0.011	88.3	--
25% Pumice	0.009	90.8	8.8
25% Perlite	0.013	86.5	-33.3
25% Ash	0.016	83.3	-64.9
25% Metakaolin	0.009	90.4	5.3
25% Shale	0.012	87.6	-22.8
25% Zeolite-1	0.012	87.2	-26.3
35% Zeolite-2	0.017	81.6	-82.5
25% Zeolite-3	0.013	86.7	-31.6
25% Fly Ash	0.010	89.9	--
Control	0.094	--	--

3.2 COMPRESSIVE STRENGTH

Many factors can have an effect on the compressive strength of concrete; while mixture proportions and aggregate properties have an effect on the overall structure and cohesiveness of concrete, incorporating pozzolanic material as a replacement for portland cement primarily effects strength by the decreasing the porosity of the interfacial transition zone (ITZ), the area between the paste matrix and the aggregate surface (Thomas, 2013). Pozzolans help by converting weak and porous calcium hydroxide, which generally forms parallel to the aggregate surface in the ITZ, to stronger and denser calcium-silicate hydrate (C-S-H) creating a stronger bond between the cement paste and aggregate. In this study, the effect of fly ash alternatives on compressive strength was evaluated using the Strength Activity Index described in ASTM C 311 (ASTM, 2011) and concrete compressive strength tested according to ASTM C 39 (ASTM, 2012).

3.2.1 ASTM C 311 Strength Activity Index

2 in. mortar cubes were made according to the procedures specified in Sections 27-30 of ASTM C 311 (ASTM, 2011). As directed by ASTM C 311 (ASTM, 2011), 20% cement by weight was replaced with the SCM to be evaluated and the water content was adjusted so that the mortar flow, measured using ASTM C 1437 (ASTM, 2007), was within $\pm 5\%$ of the control mortar mixed at a w/cm of 0.485. The mortar containing was mixed according to ASTM C 305 (ASTM, 2012) and molded and tested according to ASTM C 109 (ASTM, 2012).

3.2.2 Compressive Strength of Concrete

3.2.2.1 Concrete Mixture Design

The concrete mixture design used for all concrete studies, except ASTM C 1293 (ASTM, 2008), is shown in Table 3-6. Several factors were considered when designing

this concrete mixture. First, the w/cm was set at 0.45. This w/cm was chosen because it is commonly accepted as the maximum w/cm for load bearing structures. Next, the cement content was determined by estimating the cement paste necessary to ensure a workable mixture. The high water demand for the zeolite materials meant that the paste itself would be stiffer to begin with, and thus a relatively high cement content would be necessary. For this reason, a six-sack mix (564 lb/yd³) was found to be the most reasonable and practical cement content for the purposes of this study. Finally, the aggregate gradation was determined by the type of testing planned for this research. Because the test for CTE requires a specific coarse aggregate gradation, the gradation specified in Tex-428-A was used for all concrete mixtures. The same SCM contents used for ASTM C 1293 (ASTM, 2008) testing were used for all other concrete studies.

3.2.2.2 Mixing, Casting, Consolidation, and Curing

A naphthalene-based superplasticizer, as described in Chapter 2, was used to hit a target slump of 4 in. \pm 1 in for each concrete mixture. An initial dose based on information obtained from characterization and mortar ASR testing was estimated and added to the mixing water prior to mixing (pre-dose). If the measured slump was not within the target range, more superplasticizer was added directly to the concrete in the mixer and mixed for an additional 60 seconds (post-dose). Pre- and post-dose values for each concrete mixture are shown in Table C-1 of Appendix C. The concrete specimens used for compressive strength, drying shrinkage, RCP, and CTE testing were mixed, cast, and consolidated according to the procedures described in ASTM C 192 (ASTM, 2012). Specimens were vibrated using a vibrating table for 30-45 seconds when the measured slump was less than 3 inches and rodded according to ASTM C 192 (ASTM, 2012) if the measured slump was greater than 3 inches. After final finishing, the specimens were covered with wet burlap for

24 hours. After the specimens were removed from their molds at 24 hours, the cylinders were transferred to a moist room set to 23 °C and 100% relative humidity and the prisms used for drying shrinkage were placed in super-saturated lime water at 23 °C. Slump, air content, and unit weight were measured and recorded for every concrete mixture.

3.2.2.3 Testing

12-4 in. x 8 in. cylinders were cast for compressive strength testing at 7, 28, 56, and 90 days. At the appropriate ages, three cylinders were removed from moist storage and tested in a Forney FX-700 compression machine according to ASTM C 39 (ASTM, 2012). Neoprene pads with Shore A durometer hardness of 70 were used with metal retainers as end caps according to ASTM C 1231 (ASTM, 2012).

Table 3-6: Concrete Studies Mix Design

Component	Batch Weight lb/yd³	Weight %	Volume %⁷
Coarse Aggregate	1937	48.0	43.4
Fine Aggregate	1277	31.7	28.9
Cementitious Material	564	14.0	10.6
Water	254	6.3	15.1
Air	--	--	2.0

3.2.3 Results and Discussion

3.2.3.1 Mortar

Results for SAI testing on 2 in. mortar cubes are summarized in Table 3-7 and Figure 3-3. Table 3-7 presents the average compressive strength for each material at 7 and

⁷ These values are based on control concrete proportions. Mixture proportions for all concretes are shown in Table C-2 of Appendix C.

28 days while Figure 3-3 shows the compressive strength relative to the fly ash mortar at the same ages. The line drawn at 100% is meant to aid in comparing the mortars made with 20% SCM to 20% Fly Ash mortar.

Table 3-7: Average SAI Mortar Cube Compressive Strength
(red values indicate that the mortar failed ASTM C 618 requirements)

Mortar	w/cm	Compressive Strength, psi		Strength Relative to Control, %	
		7 day	28 day	7 day	28 day
Control	0.485	4770	5755	100.0	100.0
20% Pumice	0.505	3913	5334	82.0	92.7
20% Perlite	0.485	4109	5394	86.1	93.7
20% Ash	0.495	3422	4803	71.7	83.5
20% Metakaolin	0.495	4506	6213	94.5	108.0
20% Shale	0.500	3423	4644	71.8	80.7
20% Zeolite-1	0.560	3376	5772	70.8	100.3
20% Zeolite-2	0.570	2863	3712	60.0	64.5
20% Zeolite-3	0.640	2248	3505	47.1	60.9
20% Fly Ash	0.450	3779	4993	79.2	86.8

As stated in Chapter 2, SAI measures the reactivity of a given SCM-cement combination using a controlled flow and a variable w/cm. The ASTM C 311 (ASTM, 2011) method for SAI referenced by ASTM C 618 (ASTM, 2012) does not allow the use of water reducers or superplasticizers to reduce water demand. For this reason, it is difficult to compare the mortar results directly to the concrete results. Pumice, Perlite, Metakaolin, and Fly Ash passed ASTM C 618 (ASTM, 2012) SAI requirements at both 7 and 28 days while Ash, Shale, and Zeolite-1 passed only after 28 days of hydration. Even with the higher water contents, four materials, Pumice, Perlite, Metakaolin, and Zeolite-1, had compressive strengths greater than the fly ash mortar at 28 days or sooner. Two materials, Ash and Shale, had SAI compressive strengths only 10% lower than the Fly Ash mortars.

Zeolite-2 and Zeolite-3 did not meet ASTM C 618 (ASTM, 2012) SAI requirements (SAI $\geq 75\%$ at either 7 or 28 days) and were significantly weaker compared to the Fly Ash mortar.

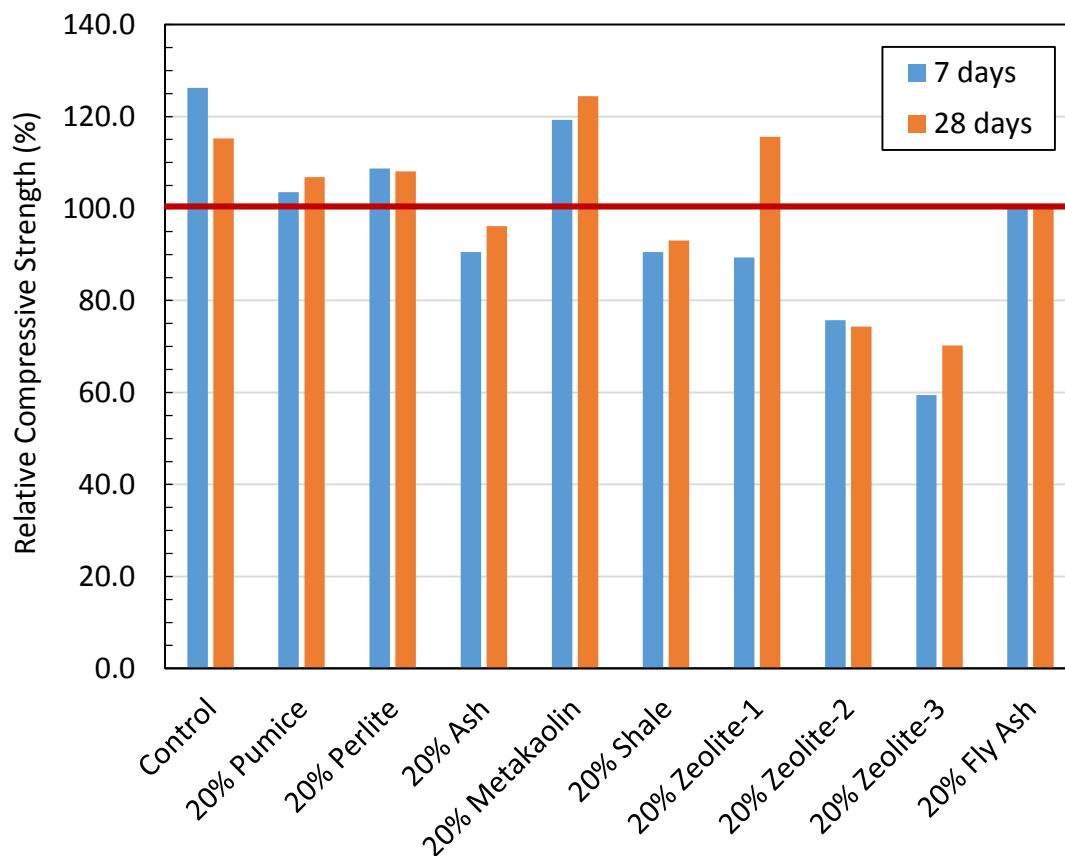


Figure 3-3: Average SAI Mortar Cube Compressive Strength Relative to Fly Ash SAI Mortar

Results in published literature for volcanic pumice, volcanic ash, and perlite generally support the results found in this research. Pumice and Ash SAI performance was similar to results published by Hossain (2005). This study found the 28 day SAI of mortars containing 20% volcanic pumice or volcanic ash from Papua New Guinea to be 83% and

94%, respectively (Hossain K. M., 2005). However, Campbell, Weise, & Love (1982) found that mortars with 20% cement replaced with volcanic ash from the 1980 Mount St. Helens eruptions decreased the compressive strength by 25-43% at an age of 28 days when compared to a control mortar, depending on where the ash was collected. The Perlite-cement combination used for this research was slightly more reactive than perlites used by Erdem et al. (2007). Their study found the SAI of two different perlites to be between 80 and 86% at both 7 and 28 days.

3.2.3.2 Concrete

Table 3-8 and Figures 3-4 and 3-5 present the average compressive strength of each concrete mixture and the compressive strength relative to the fly ash concrete. The lines drawn at 100% are meant to aid in the comparison between the concretes made with different SCMs and the concretes made with similar fly ash contents. Measured strength for all concrete cylinders tested are shown in Table D-1 in Appendix D.

This compressive strength testing shows that concrete made with both Metakaolin and Zeolite-1 performed just as well or better than concrete made with Fly Ash at a 15% cement replacement. 15% Metakaolin or 15% Zeolite-1 concrete mixtures had a 10% or less reduction in compressive strength when compared to the 15% Fly Ash concrete at all ages. Concrete made with 15% Pumice had compressive strengths similar to the 15% Fly Ash concrete at 7 and 28 days; however, at later ages, the 15% Pumice concrete showed almost 15% reduction in strength when compared to the Fly Ash concrete with the same cement replacement.

At the higher cement replacement, Pumice, Metakaolin, Shale, and Zeolite-1 performed well compared to Fly Ash. Both the 25% Metakaolin and 25% Zeolite-1 concretes had 7 and 28 day compressive strengths significantly higher than the 25% Fly

Ash concrete; the concrete with 25% cement replaced with Metakaolin had 7 and 28 day compressive strengths nearly 40% and 20% higher than the concrete made with Fly Ash at the same replacement dosage.

Table 3-8: Average Concrete Compressive Strength

Concrete Description	Compressive Strength, psi			
	7 day	28 day	56 day	90 day
Control	5650	6590	7080	7420
15% Pumice	5150	6090	6610	7050
25% Pumice	4540	6130	6710	7360
15% Perlite	4170	5350	5830	6230
25% Perlite	4150	5260	6460	6500
25% Ash	3970	5090	5810	6400
15% Metakaolin	5220	6740	7240	7450
25% Metakaolin	6350	7480	7620	7880
25% Shale	4880	6500	7370	7510
15% Zeolite-1	6240	7980	8300	8250
25% Zeolite-1	5470	7410	7350	7540
35% Zeolite-2	3190	4860	5520	5390
25% Zeolite-3	4280	5790	6390	6000
15% Fly Ash	5060	6440	7850	8100
25% Fly Ash	4590	6310	7260	7680

Perlite performed poorly at both replacement levels. Concrete with 15% Perlite showed nearly 20% reduction in strength when compared to concrete made with the same content of Fly Ash and increasing the Perlite content did not increase the compressive strength relative concrete made with the same Fly Ash content. Concrete made with 25% Ash by weight of cement also had significantly lower compressive strengths compared to concrete made with a similar Fly Ash content. The 7 and 28 day average compressive strength of concrete with 25% cement replaced with Zeolite-3 were encouraging; however, by 90 days the relative strength had decreased to less than 70% of the 25% Fly Ash

concrete. The concrete made with 35% Zeolite-2 showed strength reductions of 20-30% compared to the 25% Fly Ash concrete at all ages. Despite the poor performance of these materials, it should be noted that all concretes at all cement replacement dosages had compressive strengths greater than 4500 psi at 28 days.

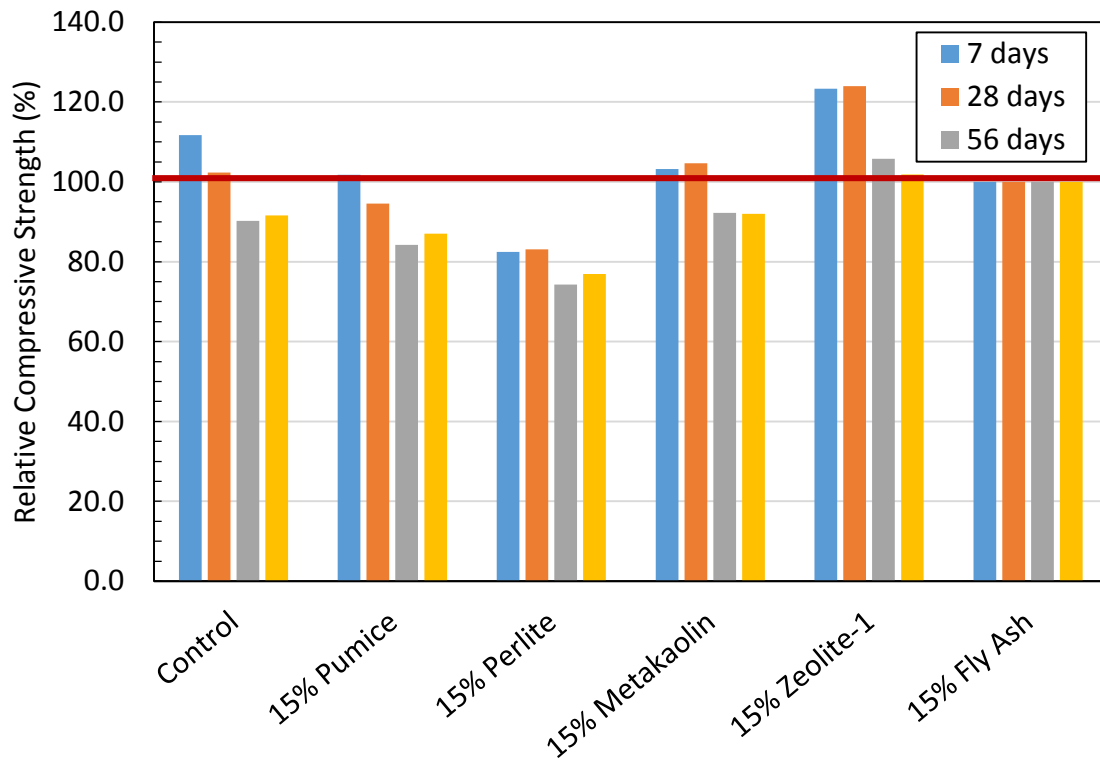


Figure 3-4: Average Concrete Compressive Strength Relative to 15% Fly Ash Concrete

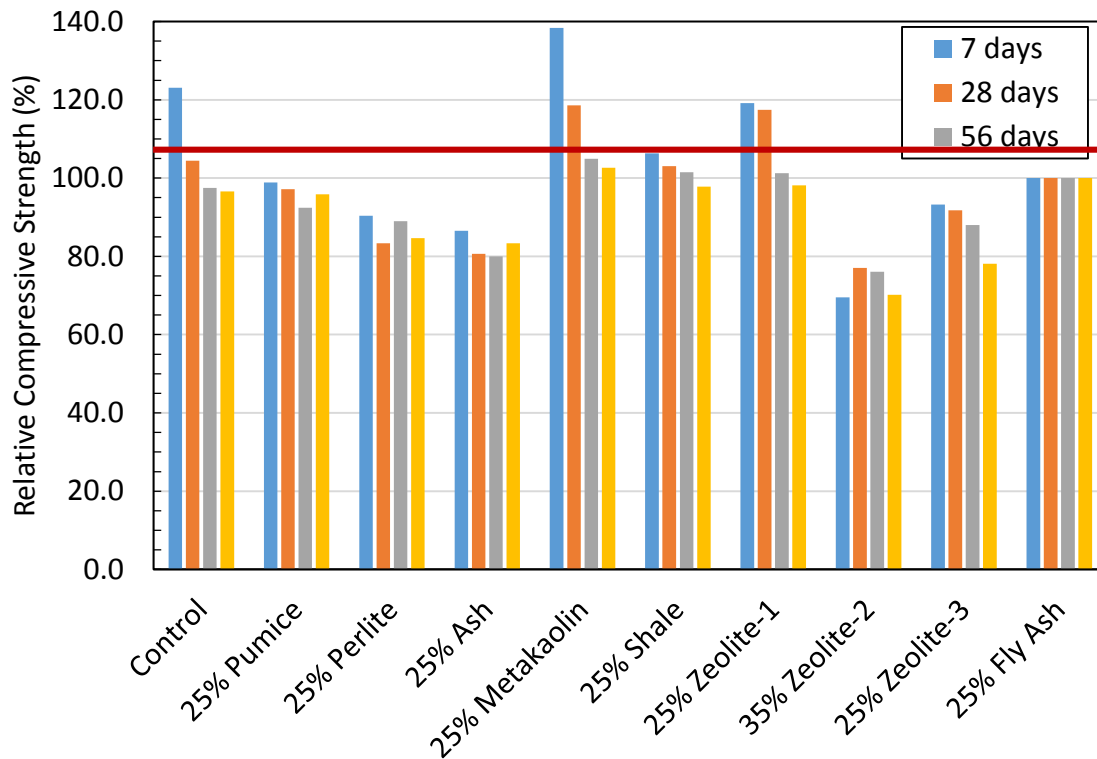


Figure 3-5: Average Concrete Compressive Strength Relative to 25% Fly Ash Concrete

A study by Hossain & Lachemi (2006) showed that concrete with 20% cement replaced with either volcanic pumice or volcanic ash from Papua New Guinea had average 28 day compressive strengths 19% and 16% lower than the control concrete. While the 25% Pumice concrete tested in this study had a 28 day compressive strength only 7% lower than the control, the 25% Ash concrete had more than 20% reduction in strength compared to the control; this may be accounted for by the difference in particle size or reactivity of the different pumice and ash sources as well as differences in mix proportions of the concrete. In a study investigating the effect of concrete containing 10-40% ground perlite by weight of cement on the compressive strength of concrete cubes, Yu, Ou, & Lee (2003) found that concrete with 15% ground perlite and a 0.30 w/cm had a compressive strength

97.9% of the control concrete after 3 days and more than 30% greater than the control after 91 days curing. These results are in stark contrast to the compressive strengths of concrete made with Perlite tested in this research; the 15% Perlite concrete had compressive strengths more than 15% lower than the control concrete at all ages. In a study on the effect of “a thermally activated alumino-silicate material” (metakaolin) on various concrete properties including compressive strength, Zhang & Malhotra (1995) showed that concrete made with 10% cement replaced with metakaolin was stronger than the control concrete at all ages. Additionally, they found that the 10% metakaolin concrete developed strength faster at early ages than concrete made with 10% cement replaced with silica fume (Zhang & Malhotra, 1995). Ahmadi & Shekarchi (2010) found that concrete with as little as 5% cement replaced with natural zeolite increased the compressive strength at all ages, and, with respect to 90 day compressive strengths, the optimum zeolite content was 15% by weight of cement; the results for concrete made with Zeolite-1 are in complete agreement. The compressive strength for concrete made with 25% Zeolite-1 by weight of cement were slightly lower than the compressive strength of concrete made with 15% Zeolite-1; however, both mixtures has compressive strengths greater than the control by 28 days.

3.3 FRESH PROPERTIES

Although rheological properties such as viscosity and yield stress provide more accurate data regarding the fresh properties of cement pastes, mortars, and concrete, mortar flow, measured by ASTM C 1437 (ASTM, 2007), and slump, measured by ASTM C 143 (ASTM, 2012), are still the most widely accepted and practical test methods in evaluating the consistency of mortars and concrete (Mehta & Monteiro, 2006). This study used these two methods in determining the water requirement of mortars made with 20% cement

replaced with SCM and the superplasticizer requirement in concrete made with similar SCM contents.

3.3.1 ASTM C 311 Water Requirement

As specified in ASTM C 311 (ASTM, 2011), the water requirement was calculated based on the water content for the SAI mortars. Water requirement was calculated by dividing the amount of water necessary to produce a mortar flow of ± 5 of the control mortar by the amount of water used in the control mortar and multiplying by 100%.

3.3.2 Concrete Slump

Concrete slump was measured according to ASTM C 143 (ASTM, 2012). As specified by this method, concrete was placed in the slump mold in three approximately equal layers and consolidated by rodding each layer 25 times with a smooth, straight steel tamping rod. After the top layer was compacted, the excess concrete was struck off and the mold was removed slowly. Slump was determined by measuring the change in height of the center of the cone of concrete to the nearest $\frac{1}{4}$ in.

3.3.3 Results and Discussion

3.3.3.1 Mortar

Figure 3-6 presents the results of the water requirement test. When the water requirement shown is greater than 100%, the material required more water than control mortar to achieve a comparable flow. As previously described, water requirement measured by test method ASTM C 311 (ASTM, 2011) is essentially a tool used to characterize a material's affinity for water. As expected, angular materials or materials with a large or charged surface area, like ground pozzolans and zeolites, attract and hold more

water than materials with round, uncharged surfaces. As previously mentioned, Yilmaz (2009) found that clinoptilolite, the primary mineral in zeolite, had a hydrophilic surface; as part of the same study, Yilmaz also found that a local fly ash had a hydrophobic surface. These surface properties may partially explain why all Zeolites had a tendency to increase the water requirement while Fly Ash required less water than the control mortar. Also, fly ash particles are spherical which have less surface area than ground, angular particles.

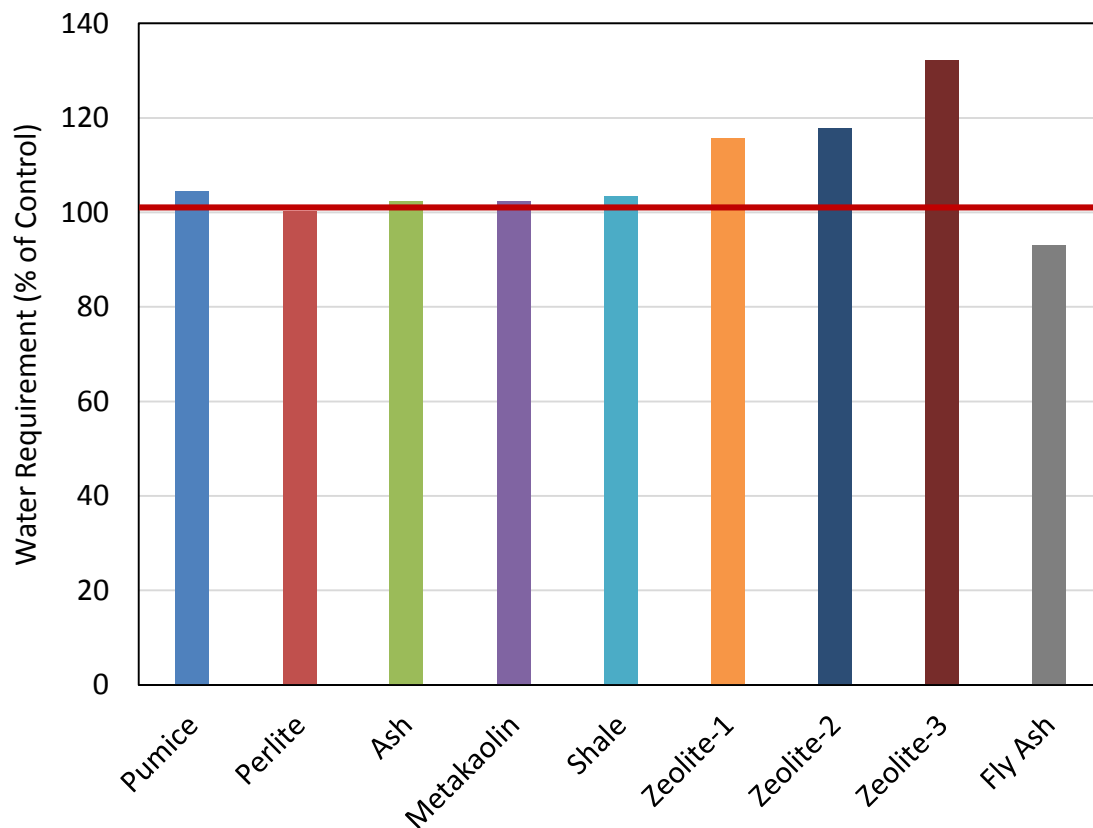


Figure 3-6: Water Requirement Based on Strength Activity Index

3.3.3.2 Concrete

Table 3-9 shows the total admixture used per concrete mixture, as a percentage of the maximum dosage, and the resulting slump. These results show that while the target slump was achievable for most concrete mixtures, one, 25% Zeolite-1, did not reach the target slump even with more than the recommended superplasticizer dosage. Additionally, although less than the maximum superplasticizer dosage was used, it was unlikely that the 25% Zeolite-3 concrete would have reached the target slump with increased superplasticizer. Admixture dosage, slump, air content, and unit weight for each concrete mixture is presented in Appendix C.

Table 3-9: Summary of Admixture Dosage and Concrete Slump Results

Concrete Description	Total Admixture, % of Max Dosage	Measured Slump, in.
Control	12.7	3.25
15% Pumice	15.5	2.50
25% Pumice	43.8	5.25
15% Perlite	9.7	3.00
25% Perlite	30.5	4.00
25% Ash	21.7	4.50
15% Metakaolin	16.2	3.50
25% Metakaolin	34.2	3.50
25% Shale	38.6	4.75
15% Zeolite-1	75.0	3.00
25% Zeolite-1	106.5	1.50
35% Zeolite-2	74.9	3.50
25% Zeolite-3	86.9	1.00
15% Fly Ash	2.2	3.75
25% Fly Ash	0.0	5.50

Although it is difficult to compare these results to those in published literature because of the significant differences in mixture design and superplasticizer type, there are significant differences between the results of this research and those of published studies. In this study, both Pumice and Ash required more superplasticizer than the control concrete to achieve a similar slump. However, Hossain & Lachemi (2006), found that concrete made with blended cement containing either 20% ground pumice or 20% volcanic ash did not significantly decrease the slump compared to a control concrete; in fact, that study found that concrete made with made volcanic ash actually increased the slump by more than 25 mm (1 in.) (Hossain & Lachemi, 2006). Zhang & Malhotra (1995) found that concrete with 10% cement replaced with metakaolin required nearly as much naphthalene-based superplasticizer as concrete made with 10% silica fume. Although silica fume was not tested as part of this research, it is widely accepted that the maximum recommended silica fume content for concrete is in the range of 8-16% because of reduced workability, even with high superplasticizer dosages (Thomas, 2013). The increased workability of the metakaolin used in this study may be attributed differences in particle size or reactivity. Slump results of this study for the zeolite concretes generally agree with results in published literature. Ahmadi & Shekarchi (2010) found that the amount of superplasticizer required to achieve a given slump was related to the zeolite content of the concrete mixture. Their control concrete required 2.7 L/m^3 superplasticizer to achieve a 65 mm (2.56 in.) slump; when the zeolite content was increased to 20% by weight of cement the concrete required more than double the superplasticizer dosage, 7.0 L/m^3 , to reach a similar slump (Ahmadi & Shekarchi, 2010).

3.4 DRYING SHRINKAGE

Strains caused by drying shrinkage can lead to cracking in finished concrete; this is not only aesthetically displeasing but can also have detrimental impacts on the durability of the affected concrete. Although cracks caused by drying shrinkage rarely propagate through the entire cross-section, even small cracks can increase the permeability of the concrete and allow troublesome ions such as sulfates and chlorides entry into the bulk matrix (Mehta & Monteiro, 2006). For this study, drying shrinkage in mortars was measured using the mixture proportions and procedure described in ASTM C 596 (ASTM, 2009). Additionally, drying shrinkage in concrete was measured according to ASTM C 157 (ASTM, 2008).

3.4.1 ASTM C 596 on Mortars

The test method for evaluating the drying shrinkage of mortars, ASTM C 596 (ASTM, 2009), was used to measure the length change of 1 in. x 1 in. x 11 ¼ in. mortar bars when exposed to 50% relative humidity. As described in the standard, the mortars contained 2 parts standard graded sand to 1 part cement and a water content such that the mortar flow measured by ASTM C 1437 (ASTM, 2007) was between 100% and 115%. After mixing and molding, the filled molds were placed in moist storage at 23 °C and 100% relative humidity. After 24 hours in moist storage, the mortars bars were removed from the molds and placed in saturated lime water for 48 hours. After this initial 3 day cure, the mortar bars were then weighed and measured using a comparator and left to air dry in a 23 °C, 50% relative humidity environmental chamber. Readings were taken after 4, 7, 11, 18, and 25 days and 8, 16, 32, and 64 weeks in the environmental chamber.

3.4.2 ASTM C 157 on Concrete

3 in. x 3 in. x 11 ¼ in. concrete prisms with gage studs at either end were cast from the same concrete mixture used for compressive strength cylinders. After curing for 24 hours under wet burlap, the prisms were removed from the molds and placed in a saturated lime water until an age of 28 days. After 28 days, the prisms were removed from the lime water, gently dried to remove any free water, and initial weight and length comparator readings were taken before the prisms were left to air dry in an environmental chamber at 50% relative humidity and 23 °C. Subsequent measurements were taken after 4, 7, 14, and 28 days and 8, 16, 32, and 64 weeks in air storage.

3.4.3 Results and Discussion

3.4.3.1 Mortar

For clarity, the available results of drying shrinkage testing on mortars are divided into three charts shown in Figures 3-7, 3-8, and 3-9. With the exceptions of Shale and Fly Ash, all SCMs tested increased the drying shrinkage of mortars as measured by ASTM C 596 (ASTM, 2009). It is important to note, however, that the different materials required a wide range of w/cm to fulfill the flow requirements of the specification. The control mortar was mixed at a 0.40 w/cm while the Fly Ash mortar was mixed at 0.385; all other SCMs required a w/cm between 0.415 and 0.505. According to the precision and bias statement of ASTM C 596 (ASTM, 2009), the acceptable difference between two test results of duplicate mixtures is 70 millionths (0.0007%); therefore, if the difference between a mortar mixture with SCM and the control mortar is less than 0.0007%, the shrinkage behavior of the two mortars can be considered the same. The only mixture to behave the same as the 20% Fly Ash mortar was the mortar with 20% Shale. Full ASTM C 596 (ASTM, 2009) results are available in tabular form in Appendix D.

Figure 3-10 and Figure 3-11 present the shrinkage and weight loss of all mortar mixtures as a function of w/cm, respectively. These figures show that the mixture w/cm accounts for a significant portion of the variation in the drying shrinkage test results. In other words, although the correlation between the weight loss of the shrinkage specimens and w/cm was more pronounced than the correlation between drying shrinkage and w/cm, w/cm still has an overwhelming effect on the results of this test.

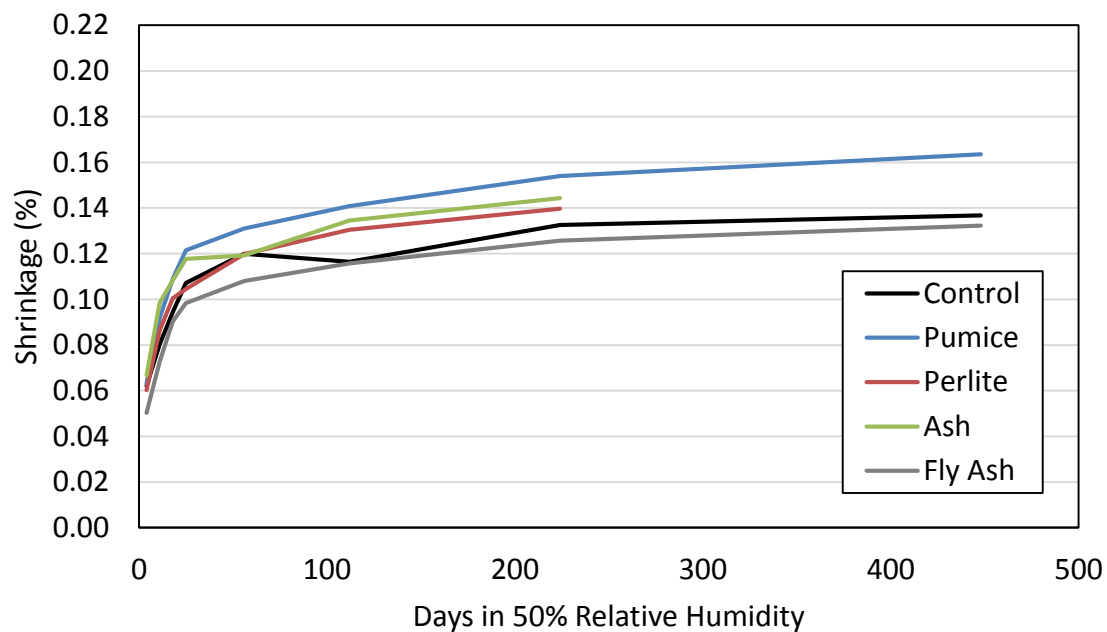


Figure 3-7: ASTM C 596 Results for Pumice, Perlite, and Ash

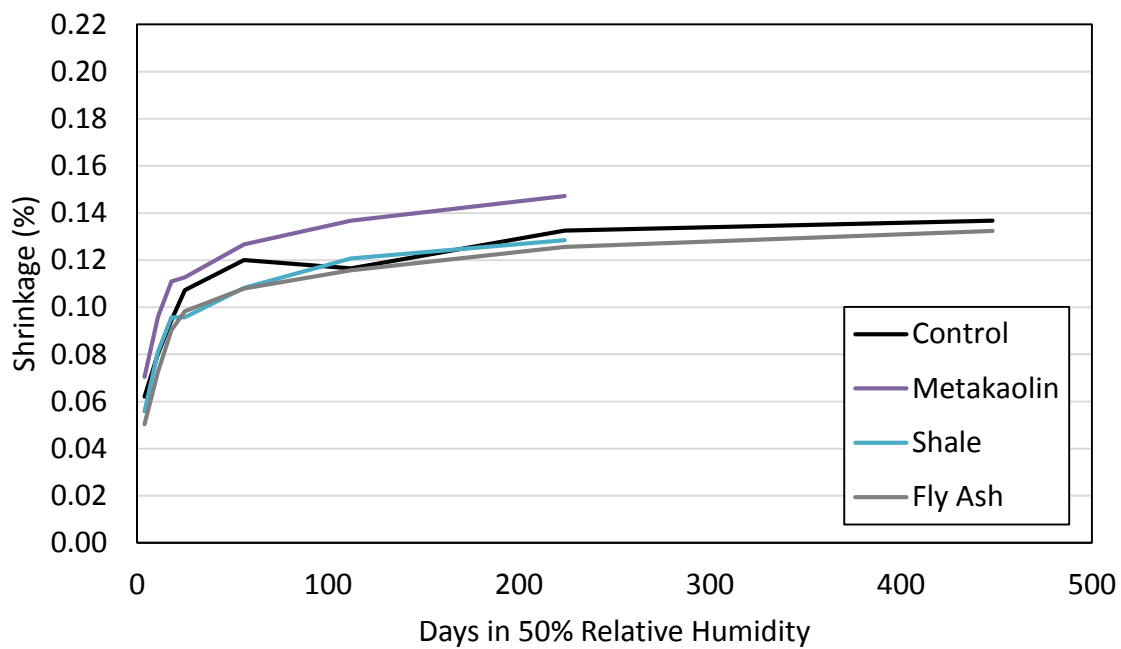


Figure 3-8: ASTM C 596 Results for Metakaolin and Shale

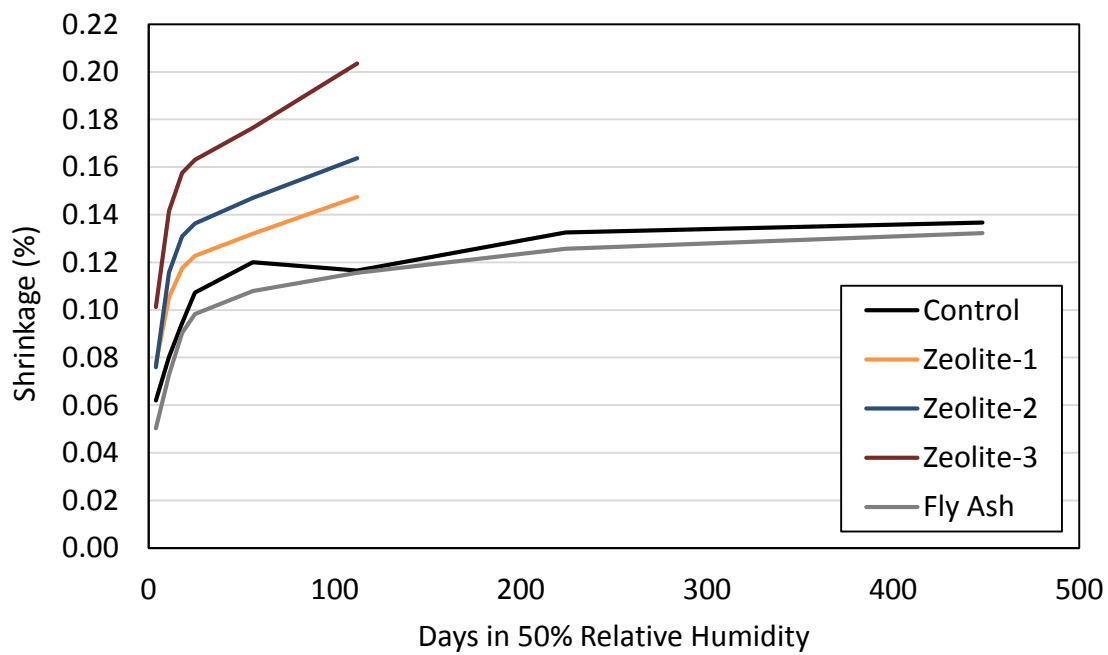


Figure 3-9: ASTM C 596 Results for All Three Zeolites

ASTM C 618 (ASTM, 2012) includes increase in drying shrinkage from a control mortar as an optional requirement for Class N pozzolans. The standard specifies that mortars made for drying shrinkage according to ASTM C 311 (ASTM, 2011) should not increase drying shrinkage strains more than 0.03% when compared to the drying shrinkage strain of the control mortar. While the mortar proportions for the control specimen specified by ASTM C 311 (ASTM, 2011) are different than the proportions used in this study for ASTM C 596 (ASTM, 2009) drying shrinkage testing, the results are worth noting. Table 3-10 presents the data relevant to the drying shrinkage criteria listed under the optional requirements of ASTM C 618 (ASTM, 2012). These results show that with the exception of Zeolite-3, all materials pass the ASTM C 618 (ASTM, 2012) optional requirement for drying shrinkage.

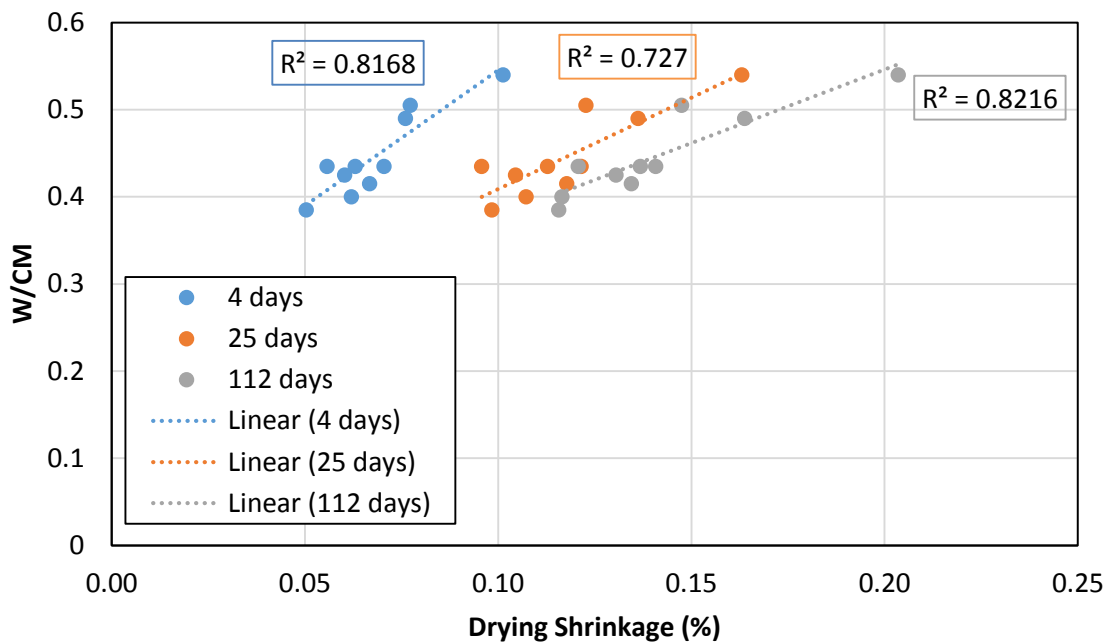


Figure 3-10: Relationship between w/cm and Drying Shrinkage

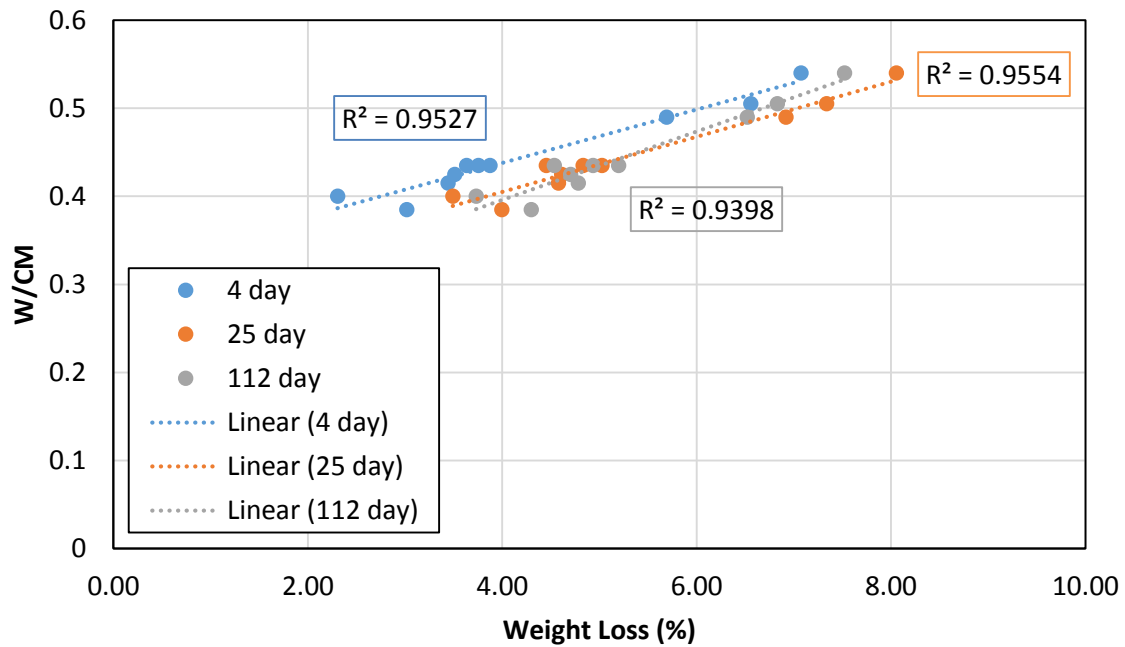


Figure 3-11: Relationship between w/cm and Weight Loss

Table 3-10: Data Relevant to ASTM C 618 Drying Shrinkage Optional Requirements

Mortar Description	Shrinkage @ 25 days, %	Difference from Control Mortar, %
Control	0.11	--
20% Pumice	0.12	0.01
20% Perlite	0.10	0.00
20% Ash	0.12	0.01
20% Metakaolin	0.11	0.01
20% Shale	0.10	-0.01
20% Zeolite-1	0.12	0.02
20% Zeolite-2	0.14	0.03
20% Zeolite-3	0.16	0.06
20% Fly Ash	0.10	-0.01

3.4.3.2 Concrete

Figures 3-12 and 3-13 show the ASTM C 157 (ASTM, 2008) drying shrinkage results relative to the shrinkage exhibited in the 15% and 25% fly ash concretes respectively. Full results for ASTM C 157 (ASTM, 2008) testing are presented in tabular form in Appendix D. The precision and bias statement of ASTM C 157 (2008) specifies that the dryings shrinkage results from duplicate concrete mixtures should not differ by more than 0.0137%. In other words, mixtures with averages that are within 0.0137% of each other can be considered to have the same drying shrinkage. With this in mind, the concrete made with 15% fly ash alternatives had measured drying shrinkages similar to the 15% Fly Ash concrete; also, the 15% SCM concretes did not differ significantly from the control at any time. Three high SCM concrete mixtures, 25% Metakaolin, 25% Shale, and 25% Zeolite-1, did not differ significantly from either the 25% Fly Ash concrete or the control concrete at any time. However, five SCM concrete mixtures, 25% Pumice, 25% Perlite, 25% Ash, 35% Zeolite-2, and 25% Zeolite-3, had measured drying shrinkage that differed significantly from the control concrete beginning after 28 days in 50% relative humidity. Also, the 25% Pumice, 25% Perlite, 35% Zeolite-2 and 25% Zeolite-3 concrete mixtures had drying shrinkage readings significantly different than the 25% Fly Ash concrete beginning with the 28 day reading. This was contrary to some results found in published literature; three studies on three different materials, volcanic pumice, volcanic ash, and metakaolin found the drying shrinkage of concrete made with these materials as cement replacements was not significantly different from their respective control concretes. Hossain et al. (2011) studied the effect of volcanic pumice and volcanic ash on the drying shrinkage of concrete. They found that concrete made with 20% ground pumice by weight of cement showed slightly less drying shrinkage than a control concrete after 12 weeks while concrete made with 20% volcanic ash showed slightly more drying shrinkage that

the control in the same time period (2011); however, the difference in drying shrinkage from the control was not significant according to the precision and bias statement of ASTM C 157 (ASTM, 2008). Similarly, both Zhang & Malhotra (1995) and Guneyisi, Gesoglu, & Mermerdas (2008) found that concretes containing metakaolin as an SCM experienced slightly less drying shrinkage compared to a control concrete. Again, however, this reduction was within the standard of error of ASTM C 157 (ASTM, 2008).

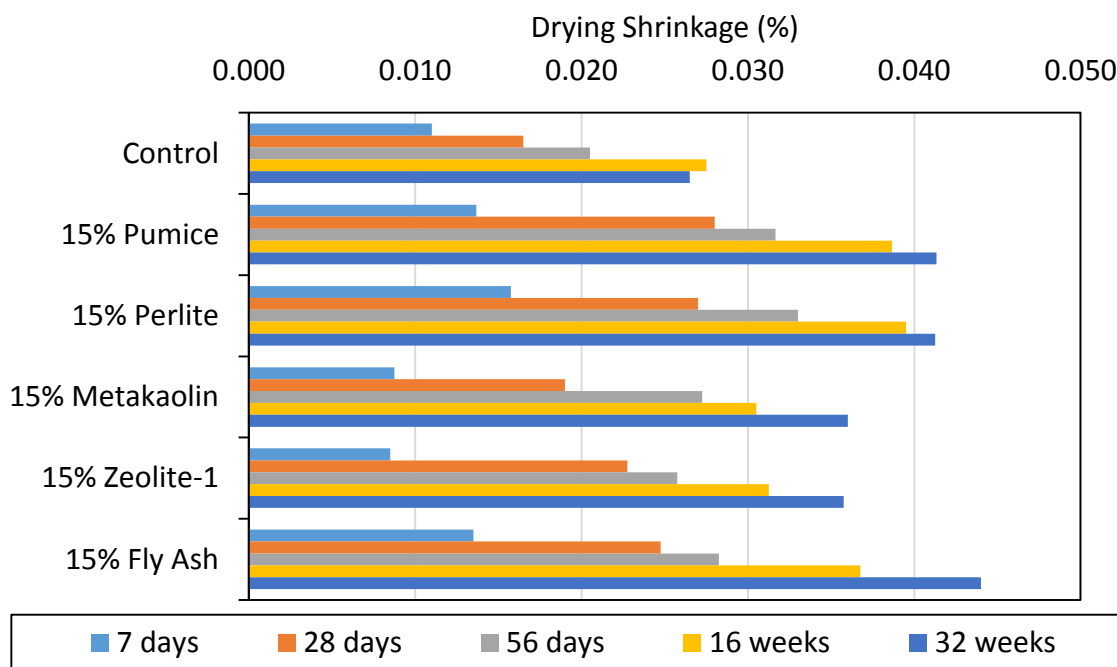


Figure 3-12: ASTM C 157 Drying Shrinkage for 15% SCM Concretes

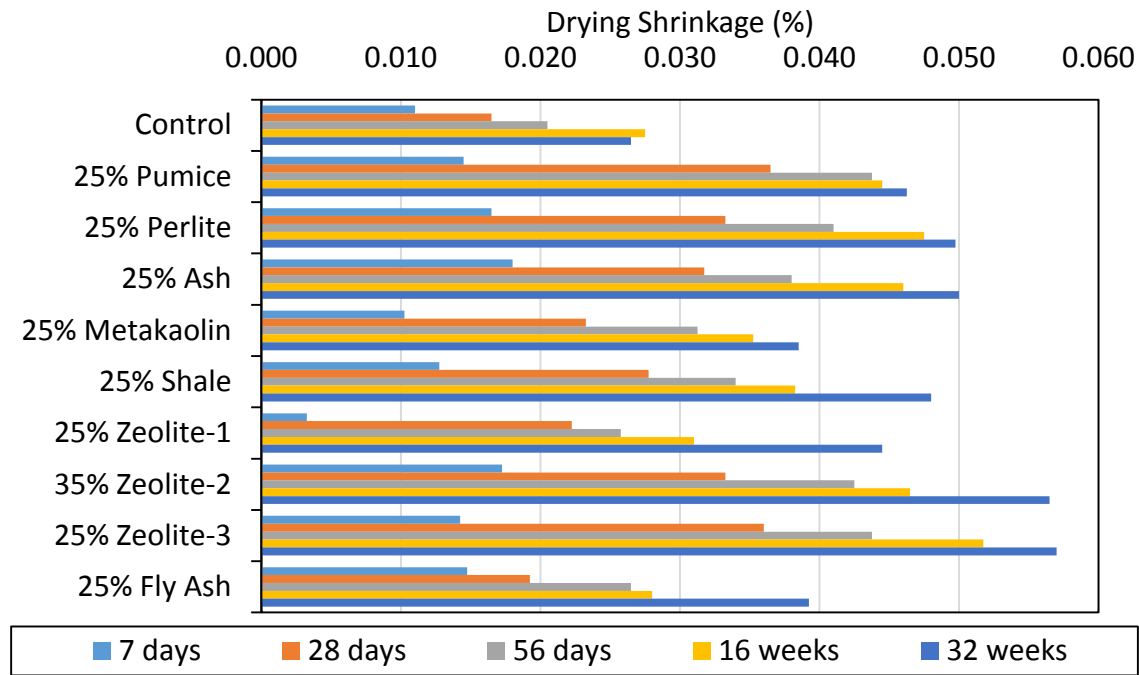


Figure 3-13: ASTM C 157 Drying Shrinkage for 25% and 35% SCM Concrete

3.5 SULFATE EXPANSION

Expansion and deterioration of concretes exposed to sulfates in soils or groundwater have been observed for many years. The primary preventive measures used to combat this reaction are the use of sulfate resistant cement, Type II or Type V, a low w/cm, and appropriate SCM contents when necessary (American Concrete Institute, 2008). This study evaluated the sulfate resistance of mortars made with the same SCM contents used in the concrete studies to determine if each SCM adequately suppressed expansion due to sulfate attack.

3.5.1 Test Method

ASTM C 1012 (ASTM, 2013) is the considered the benchmark test for determining the susceptibility of a given cement-SCM combination to resist chemical attack due to

exposure to sodium sulfate solution. As specified by this method, six - 1 in. x 1 in. x 11 ¼ in. mortar bars with gage studs at each end were prepared from a mortar containing 2.75 parts standard graded sand to 1 part cementitious material. The w/cm for the control was 0.485 and the water content for SCM containing mortars was such that the mortar flow measured by ASTM C 1437 (ASTM, 2007) was within $\pm 5\%$ of the control mortar. Six - 2 in. mortar cubes were also prepared from the same mortar mixture. The filled mortar bar and cube molds were then sealed and submerged in a water bath set to 38 °C to accelerate curing. After 24 hours, molded specimens were removed from the water bath and the specimens were removed from the molds. Immediately after removal from the molds, the compressive strength of two mortar cubes was tested. If the compressive strength was less than 2850 psi, the mortar bars were placed in saturated lime water with the remaining mortar cubes until the average compressive strength of two mortar cubes reached 2850 psi. When the average compressive strength reached 2850 psi, the mortar bars were measured using a comparator and placed in a 5% sodium sulfate solution for testing. Subsequent readings were taken at 1, 2, 3, 4, 8, 13, and 15 weeks and at 4, 6, 9, and 12 months.

3.5.2 Results and Discussion

Essentially, two sets of specimens were evaluated: one set made strictly according to ASTM C 1012 (ASTM, 2013), with a constant flow, and a second set with w/cm in the range of 0.50 ± 0.01 . When the water content required to achieve the flow specified by ASTM C 1012 (ASTM, 2013) was outside of the w/cm range of 0.50 ± 0.01 , additional mortars with a w/cm of 0.51 were mixed using the same naphthalene superplasticizer used for the concrete mixtures when necessary. Control, 15% Fly Ash, and 25% Fly Ash mortars were also mixed at a w/cm of 0.51 for comparison. Figures 3-14, 3-15, 3-16, and 3-17 show the results of ASTM C 1012 (ASTM, 2013) testing.

With the exception of the Zeolite-2 and Zeolite-3, mortars made according to the standard, the measured expansion for all mortars are within the error of the test as specified by the precision and bias statement of ASTM C 1012 (ASTM, 2013). Mortars made with 35% cement replaced with Zeolite-2 and 25% cement replaced with Zeolite-3 had expansions similar to all other mortars until 8 weeks; between 4 and 8 weeks the expansion for these two mortars increased from less than 0.05% to more than 0.40%.

According to the sulfate resistance requirements of ASTM C 618 (ASTM, 2012), after 6 months of testing according to ASTM C 1012 (ASTM, 2013), mortars with expansions less than 0.10% are considered suitable for moderate sulfate exposure while mortars with expansions less than 0.05% are suitable for high sulfate environments. Based on these criteria, mixtures containing 15% Pumice, Perlite, or Zeolite-1 or 25% Pumice, Perlite, Metakaolin, or Zeolite-1 can be considered suitable for high sulfate environments.⁸ Although the testing for the 25% Shale mortar and all four Fly Ash mortars are not completed up to 6 months, the data available indicate that the mixture is on track to meet the 0.05% expansion limit at 6 months. Both the control mixture mixed according to ASTM C 1012 (ASTM, 2013) and the control mixed at the higher w/c surpassed the high sulfate resistance cut-off by the 15th week of testing; the ASTM C 1012 (ASTM, 2013) control mixture surpassed the moderate sulfate resistance cut-off between the 4 and 6 month readings. Surprisingly, the 15% Metakaolin mortar only had two intact mortar bars at the 6 month reading, meaning the mixture failed the test completely. The Zeolite-2 and Zeolite-3 mortars mixed according to ASTM C 1012 (ASTM, 2013) failed completely at 4 months and 13 weeks, respectively, with no mortars left intact. Also, the Zeolite-2 and Zeolite-3

⁸ This statement refers to the 15% and 25% Zeolite-1 mortars mixed according to ASTM C 1012 (ASTM, 2013)

mortars mixed with a 0.51 w/c will not meet ASTM C 618 (ASTM, 2012) criteria for sulfate resistance as both had expansions greater than 0.10% at 4 months of testing.

Although it is still too early to definitively say that the materials used in this research are effective in controlling expansion due to sulfate exposure, examples from published literature are encouraging. Khatib & Wild (1998) used a method similar to ASTM C 1012 (ASTM, 2013) to study the sulfate resistance of mortars made with 5-25% metakaolin. They found that when a cement with an intermediate C_3A content (7.8%) was used only 10% metakaolin by weight of cement was necessary to limit expansion to 0.10% after 18 months exposure to 5% sodium sulfate solution; however, when cement with a high C_3A content (11.7%) was used, 20% metakaolin by weight of cement was required to keep expansion below 0.10% after 18 months in sodium sulfate solution (Khatib & Wild, 1998). Karakurt & Tapcu (2011) evaluated the sulfate resistance of mortars containing 30% natural zeolite using ASTM C 1012 (ASTM, 2013), except that 10% sodium sulfate solution was used. They found that after 6 months immersion in 10% sodium sulfate solution, the control mortar experienced an expansion greater than 0.14% while the 30% zeolite mortar measured less than 0.02% expansion. According to ASTM C 1157 (ASTM, 2011), the mortar made with 30% cement replaced with zeolite has the equivalent performance as a mortar with a Type HS (high sulfate resistance) cement.

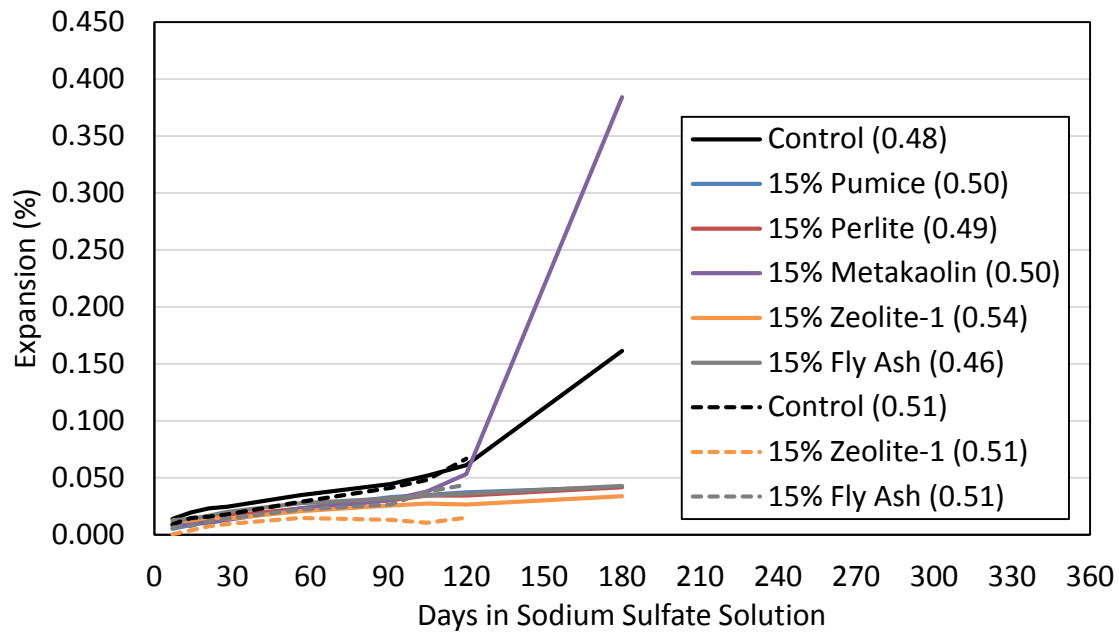


Figure 3-14: ASTM C 1012 Results for Low SCM Content Mortars
(numbers in parenthesis indicate w/cm)

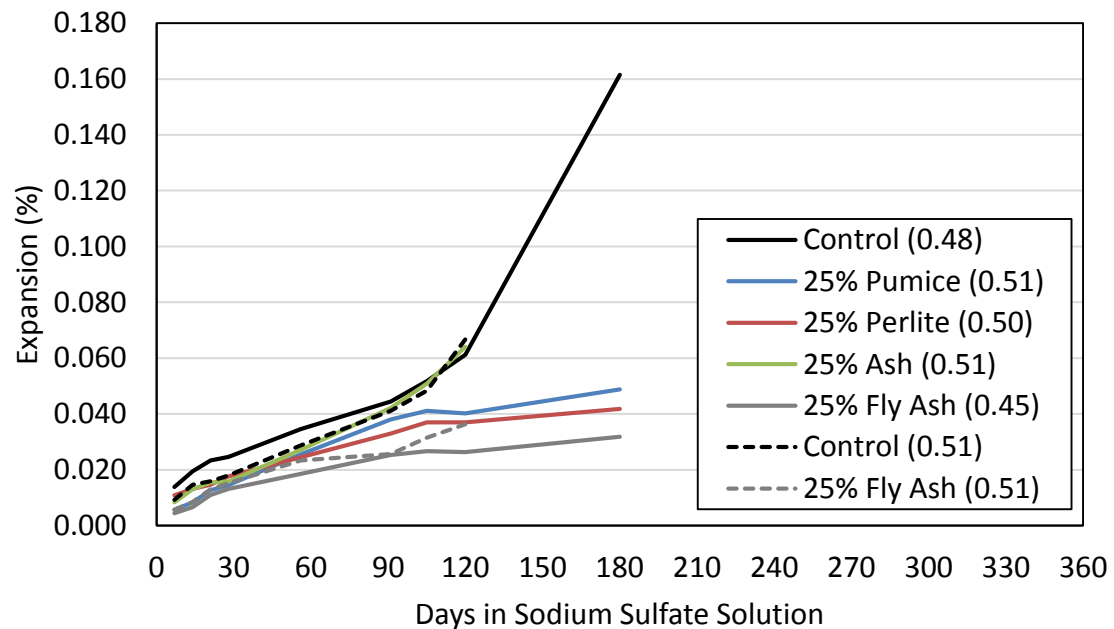


Figure 3-15: ASTM C 1012 Results for Mortars Made with 25% Pumice, Perlite, and Ash
(numbers in parenthesis indicate w/cm)

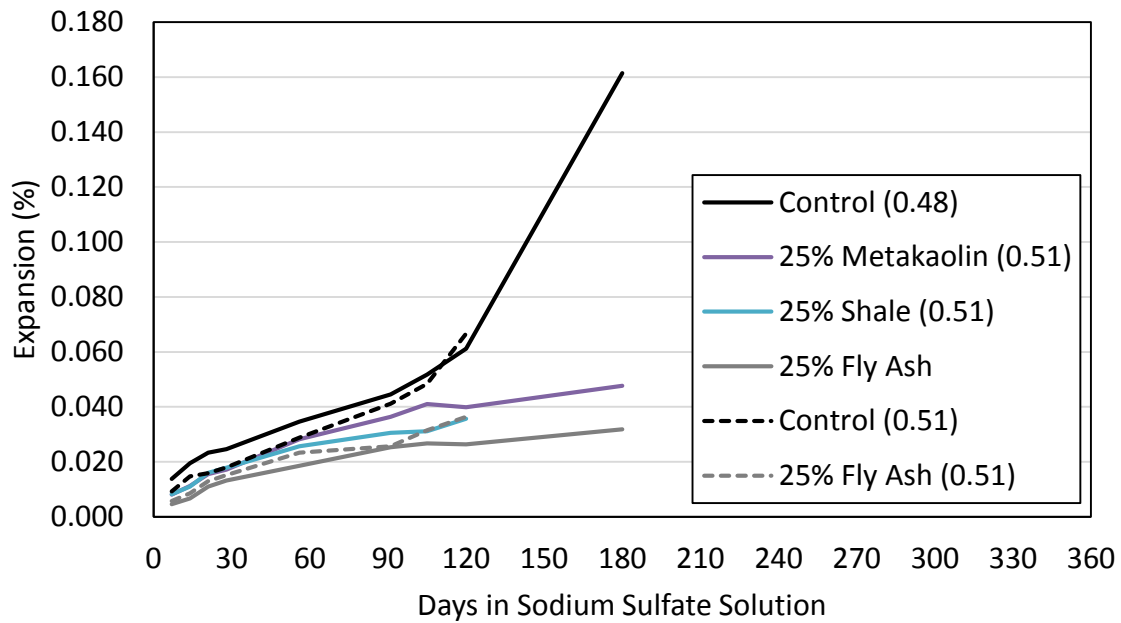


Figure 3-16: ASTM C 1012 Results for Mortars Made with 25% Metakaolin and Shale (numbers in parenthesis indicate w/cm)

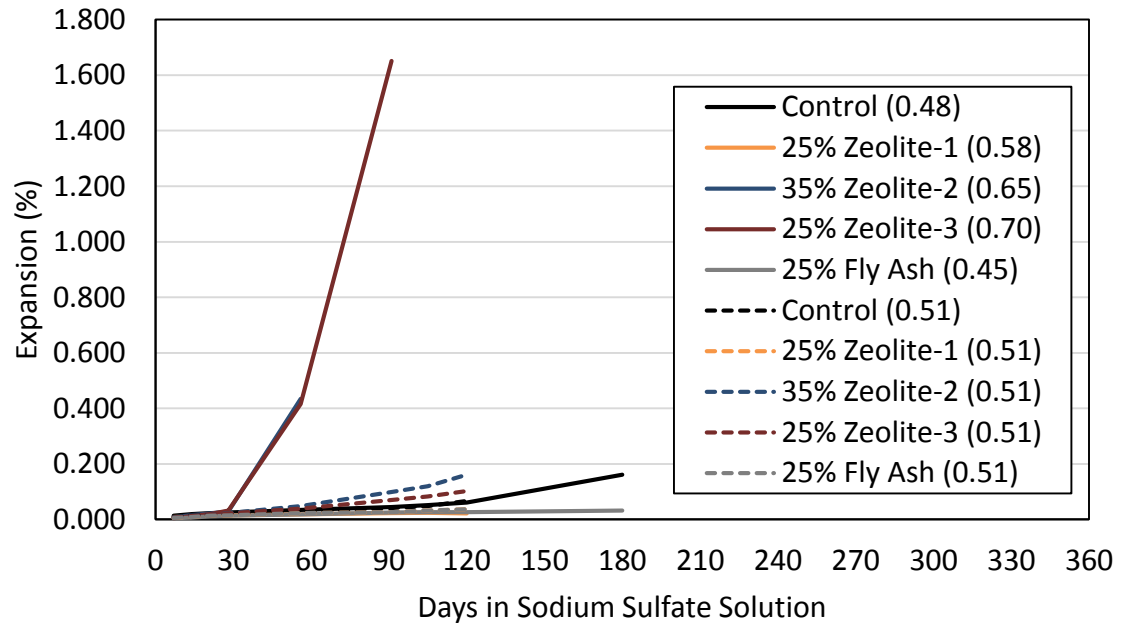


Figure 3-17: ASTM C 1012 Results for Mortars Made with 25% Zeolite-1 and Zeolite-3 and 35% Zeolite-2 (numbers in parenthesis indicate w/cm)

3.6 RAPID CHLORIDE PENETRABILITY (RCP)

3.6.1 Test Method

Rapid chloride penetrability of concrete cylinders was measured according to ASTM C 1202 (ASTM, 2012). As this method specifies, 4 in. x 8 in. cylinders were cut into 2 in. thick slices and conditioned in a vacuum desiccator. After conditioning, the 2 in. slices were sealed in the test set-up, shown in Figure 3-18, using rubber gaskets on either end of the slice to achieve a good seal. Once assembled, one side of the test cell was filled with a 3% NaCl solution and the other side was filled with a 0.3 N NaOH solution. The test cell was then connected to a 60 V power supply. Once the power supply was turned on an initial current reading was taken and additional readings were taken every 30 minutes for 6 hours. The total charge passed through the test specimen was determined by finding the area under the current-time curve and adjusting the value for a 4 in. diameter cylinder.

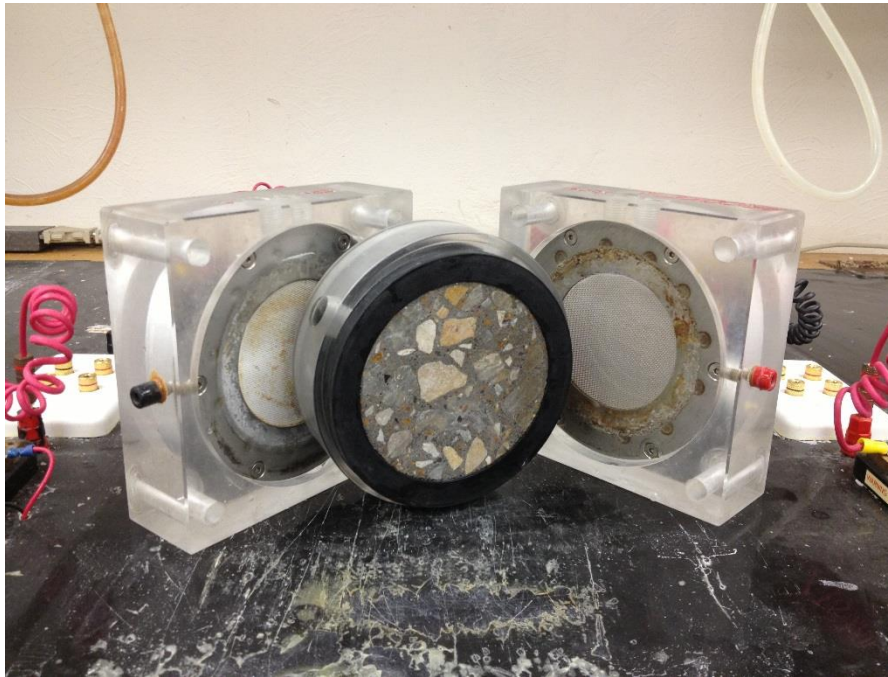


Figure 3-18: Rapid Chloride Penetrability Test Set Up

3.6.2 Results and Discussion

RCP was measured at 70 and 224 days for the Control, 15% Pumice, 15% Perlite, 15% Metakaolin, and 15% Zeolite-1 concrete mixtures; these results are shown in Figure 3-19. For all other concrete mixtures, RCP was only measured at 260 days, shown in Figure 3-20. The results presented in Figure 3-19 show that despite only a 20% reduction in chloride ion penetrability in the control concrete from 70 to 224 days, both the 15% Pumice and 15% Perlite concretes reduced chloride ion penetrability by 46% and 54%, respectively. This indicates that there is still a significant amount of hydration or densification of hydration products occurring during this time period. Although not as significant, the 15% Metakaolin and 15% Zeolite-1 concretes also showed a decrease in chloride ion penetrability from 70 to 224 days. With the exception of the control concrete, all concrete mixtures tested had very low chloride ion penetrability according to thresholds stated in ASTM C 1202 (ASTM, 2012)

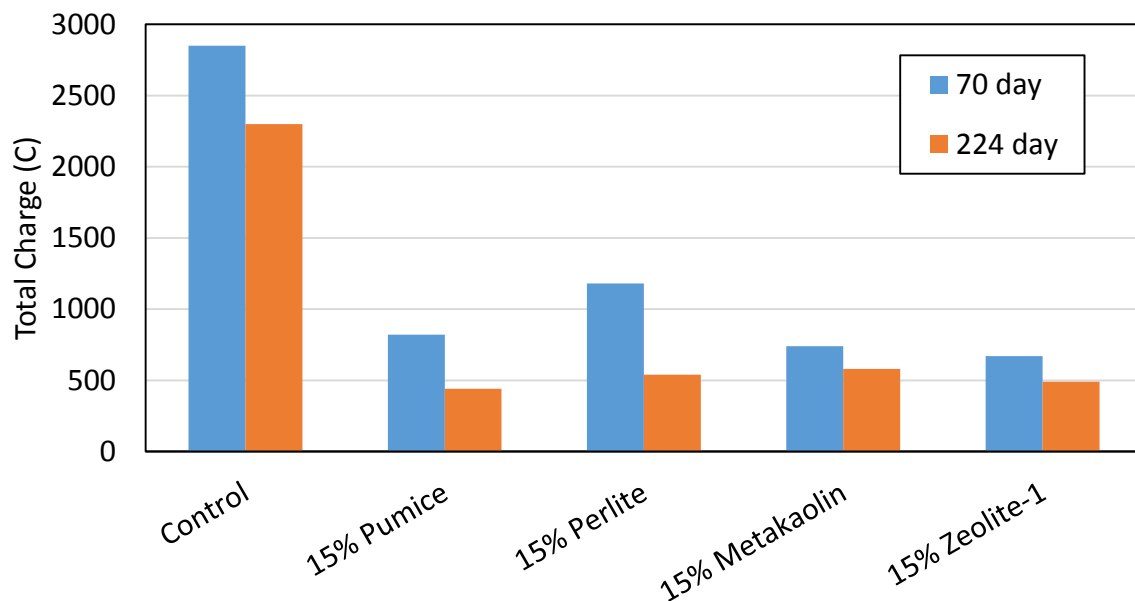


Figure 3-19: Rapid Chloride Penetrability for Low SCM Concretes

The results presented in Figure 3-20 show that while the concrete made with cement replaced with SCMs had lower chloride ion penetrability than the control concrete, the performance of the fly ash alternatives was similar to that of fly ash. The 15% SCM concretes reduced chloride ion penetrability on average by approximately 77% compared to the control concrete. Concretes made with a higher SCM content reduced chloride ion penetrability on average by approximately 79%. Additionally, although the difference between concretes made with 15% and 25% cement replaced with SCM was not significant, increasing the SCM content did decrease the chloride ion penetrability for each SCM.

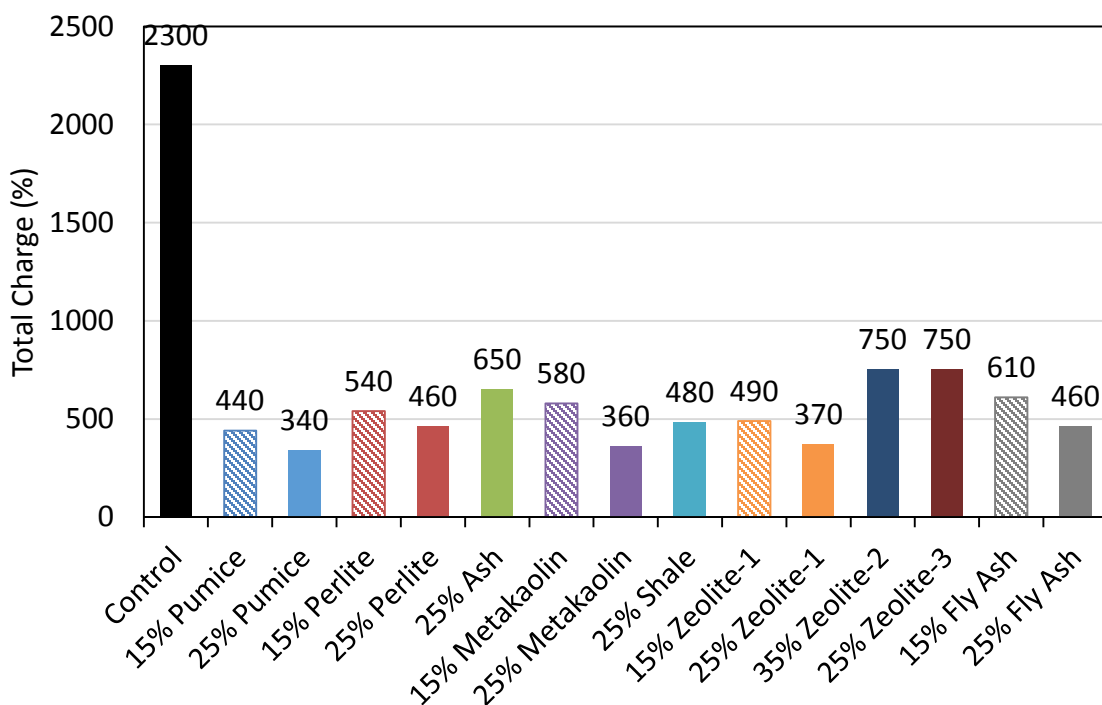


Figure 3-20: Rapid Chloride Penetrability of All Concrete Mixtures Measured at 224 days

Previous studies on pumice, volcanic ash, perlite, and metakaolin have indicated that all four materials either decrease the chloride ion penetrability or are capable of producing low permeability concrete. Hossain & Lachemi (2006) found that concrete with a w/cm of 0.45 and 20% cement replaced with either ground pumice or volcanic ash reduced the chloride ion penetrability, as measured by ASTM C 1202 (ASTM, 2012), by 19% and 23%, respectively, at an age of 56 days. Additionally, in concrete with a w/cm of 0.35 and 20% cement replacement with ground pumice or volcanic ash, the total charge passed was reduced by 16% and 19%, respectively (Hossain & Lachemi, 2006). Zhang & Malhotra (1995) found that concrete made with 10% metakaolin was similar to 10% silica fume concrete in chloride ion penetrability as measured by ASTM C 1202 (ASTM, 2012); they found that the total charge passed in the 10% metakaolin concrete was more than 80% lower than that of the control mixture at both 28 and 90 days (Zhang & Malhotra, 1995). While studying high volume natural pozzolan concretes, Uzal, Turanli, & Mehta (2007) found that using perlite in concrete provides fairly good resistance to chloride ion penetration. Using ASTM C 1202 (ASTM, 2012), they found that total charge passed in concrete made with 50% perlite by weight of cement was only 684 coulombs after 91 days of curing. This means the 50% perlite concrete had a “very low permeability” rating based on the rating system in ASTM C 1202 (Uzal, Turanli, & Mehta, 2007; ASTM, 2012).

3.7 COEFFICIENT OF THERMAL EXPANSION (CTE)

CTE of concrete is important when considering the performance and durability of concrete pavement. In plain concrete pavements, high CTE concrete may cause early-age cracking, curling, faulting, and joint spalling (Crawford, Gudimettla, & Tanesi, 2010). Also, high CTE concrete may increase the crack spacing and crack width in continuously reinforced concrete pavements, affecting the crack load transfer efficiency (Mallela, et al.,

2005). CTE is primarily dominated by the aggregate type and source, and other factors including SCM type and content have smaller effects on the CTE value (Naik, Kraus, & Kumar, 2011). However, because the effect on the CTE value of concrete containing these fly ash alternatives is not well established, it was deemed important to investigate what effect, if any, these materials had on the CTE value of concrete used in this research.

3.7.1 Test Method

According to Tex-428-A, two 4 in. x 8 in. concrete cylinders were cut to a length of 7 in. \pm 0.1 in. and submerged in water for 48 hours. The cut cylinders were then measured to the nearest 0.001 in. using a caliper and submerged in temperature-controlled water baths programmed to cycle between 10 °C and 50 °C. Inside the water baths the cylinders were placed in testing frames equipped with a differential variable reluctance transformer (DVRT) used to measure the change in length of the specimen. After completing three cycles the specimens were removed and the data were analyzed using an Excel spreadsheet provided by TxDOT⁹.

3.7.2 Results and Discussion

CTE results are summarized in Table 3-11. The table presents the average CTE value for each of two cylinders as well as the difference between the overall average for a given concrete mixture and the overall average of the control concrete. Although the results indicated by an asterisk were not within the precision specified by the standard, specimens that were tested until the CTE results were in compliance with the standard did not differ significantly from the original CTE value.

⁹ The Excel spreadsheet used for analysis was created by Jerry Peterson at TxDOT

To the author's knowledge, there are no published data on the effect of these SCMs on the CTE of concrete. Therefore, these data cannot be compared to published literature.

Table 3-11: Coefficient of Thermal Expansion Results for Selected Concrete Mixtures

Concrete Description	Cylinder 1 μ -strain/ $^{\circ}$ F	Cylinder 2 μ -strain/ $^{\circ}$ F	Average μ -strain/ $^{\circ}$ F	Difference from Control μ -strain/ $^{\circ}$ F
Control	3.61	3.56	3.59	
25% Pumice	*4.16	4.15	4.16	0.57
25% Ash	*4.19	3.94	4.07	0.48
25% Metakaolin	*4.22	3.99	4.11	0.52
25% Shale	4.22	4.01	4.12	0.53
25% Zeolite-1	4.36	4.00	4.18	0.60
25% Fly Ash	4.06	*3.56	3.81	0.23

Chapter 4: Conclusions

4.1 SUMMARY OF PROJECT

As the supply of high quality fly ash is threatened by impending environmental and health and safety regulations, the need for finding suitable and reliable pozzolanic alternatives grows more urgent. In this research, eight natural, lightly processed fly ash alternatives were evaluated to determine their appropriateness for use as pozzolans in portland cement concrete. These materials were compared to the criteria set forth by ASTM C 618 (ASTM, 2012) for Class N natural pozzolans and used in mortar and concrete to evaluate their propensity for mitigating deleterious chemical reactions and their effect on various fresh and hardened concrete properties.

4.2 CONCLUSIONS

The following sections describe the conclusions that can be drawn from each of the tests conducted for this research.

4.2.1 SCM Characterization

- All of the natural materials tested, with the exception of the zeolites, meet all ASTM C 618 (ASTM, 2012) requirements for Class N pozzolans.
- Coarser natural pozzolans, such as Zeolite-2 and Zeolite-3, did not meet reactivity criteria and may require more processing to be pozzolanically suitable for use in portland cement concrete.

4.2.2 Alkali Silica Reaction

- According to ASTM C 1260/C 1567 (ASTM, 2007; ASTM, 2013) test results, all materials tested are capable of reducing expansion due to ASR at cement replacement percentages comparable to fly ash.

- After 9 months of testing there are no definitive trends with regard to SCM content and expansion due to ASR however, all concretes containing SCMs reduced expansion significantly compared to the control concrete.
- After 9 months of testing, concretes made with 15% Perlite, 15% Metakaolin, 15% Zeolite-1, 25% Pumice and 25% Metakaolin reduced expansion comparable to their respective Fly Ash concrete mixtures.

4.2.3 Compressive Strength

- ASTM C 311 (ASTM, 2011) SAI testing indicated that all materials except Zeolite-2 and Zeolite-3 meet ASTM C 618 (ASTM, 2012) reactivity requirements for Class N pozzolans.
- Pumice, Perlite, Metakaolin, and Zeolite-1 all had compressive strengths similar to or greater than the fly ash mortar.
- At the 15% cement replacement level, only Metakaolin and Zeolite-1 had compressive strengths at all ages comparable to the Fly Ash concrete with the same cement replacement.
- At the 25% cement replacement level, only Metakaolin, Shale, and Zeolite-1 had compressive strengths at all ages comparable to the Fly ash concrete with the same cement replacement.

4.2.4 Fresh Properties

- ASTM C 311 (ASTM, 2011) water requirement testing showed that all fly ash alternatives tested significantly increased the amount of water needed to achieve the desired mortar flow when compared to the Fly Ash mortar.

- The admixture dosages required to achieve the target slump range indicate that the zeolites may cause workability problems in mixtures requiring a moderate to high slump.

4.2.5 Drying Shrinkage

- Results from mortar testing indicate that all fly ash alternatives increase drying shrinkage in mortars made with variable w/c and constant flow.
- In concretes, all SCMs, including Fly Ash, increased drying shrinkage at nearly every age.

4.2.6 Resistance to Sulfate Attack

- As little as 15% cement replacement with Pumice, Perlite, or Zeolite-1 is adequate to limit expansion due to sulfate attack in severe exposure conditions.
- Mortars with 15% Pumice, Perlite, or Zeolite-1 or 25% Pumice, Perlite, Ash, Metakaolin, Shale, or Zeolite-1 were virtually indistinguishable from mortars with Fly Ash at similar cement replacement dosages.
- The coarser zeolites, Zeolite-2 and Zeolite-3 were incapable of creating sulfate resistant mortar.

4.2.7 Rapid Chloride Penetrability

- Testing indicated that as little as 15% and 25% cement replacement with these fly ash alternatives dramatically reduced the chloride ion penetration as measured by ASTM C 1202 (ASTM, 2012); the chloride ion penetrability

decreased from “moderate” in the control concrete to “very low” in all concretes made with SCMs.

4.2.8 Coefficient of Thermal Expansion

- CTE testing indicates that these fly ash alternatives, in the condition and proportions utilized in this research, do not significantly alter the thermal expansion properties of concrete compared to both a control concrete and concrete made with Fly Ash at similar cement replacements.

4.3 RECOMMENDATION AND SUGGESTIONS FOR FUTURE WORK

Based on the results of the testing presented in this thesis, the author presents the following recommendation and suggestions for future work:

1. The Pumice, Perlite, Ash, Metakaolin, Shale, and Zeolite-1 used in this study are suitable for use as pozzolans in portland cement concrete so long as suitable admixtures are utilized to minimize the impact of the increased water demand of these materials.
2. The effect of particle size on the reactivity and water requirement of mortars and concretes containing zeolites may help explain why the finer zeolite, Zeolite-1, performed better than both coarser zeolites in all aspects of this research.
3. A study of mortars and concretes made with an inert powder with an average particle size similar to the materials used in this research would help determine if the benefits to strength and chloride ion penetrability are a result of a pozzolanic reaction or denser particle packing.
4. A more detailed look at the availability and costs associated with each of these materials would help determine if any of these natural materials are actually

capable of replacing fly ash as a “green”, cost effective supplementary cementitious material.

Appendix A: ASTM C 1260/ASTM C 1567 Results

Table A-1: Admixture Dosages for Each SCM Content Tested
(red values indicate the required dosage exceeded the recommended maximum dosage)

Material	Admixture Dosage (mL/100 kg cement)					
	10% SCM	15% SCM	20% SCM	25% SCM	30% SCM	35% SCM
Pumice	124	155	127	X	X	X
Perlite	124	155	127	X	X	X
Ash	X	X	127	139	X	X
Metakaolin	124	155	183	X	X	X
Shale	X	X	124	155	X	X
Zeolite-1	341	511	651	X	X	X
Zeolite-2	X	X	356	806	961	1348
Zeolite-3	X	X	1116	1426	X	X
Fly Ash	X	X	0	X	X	X

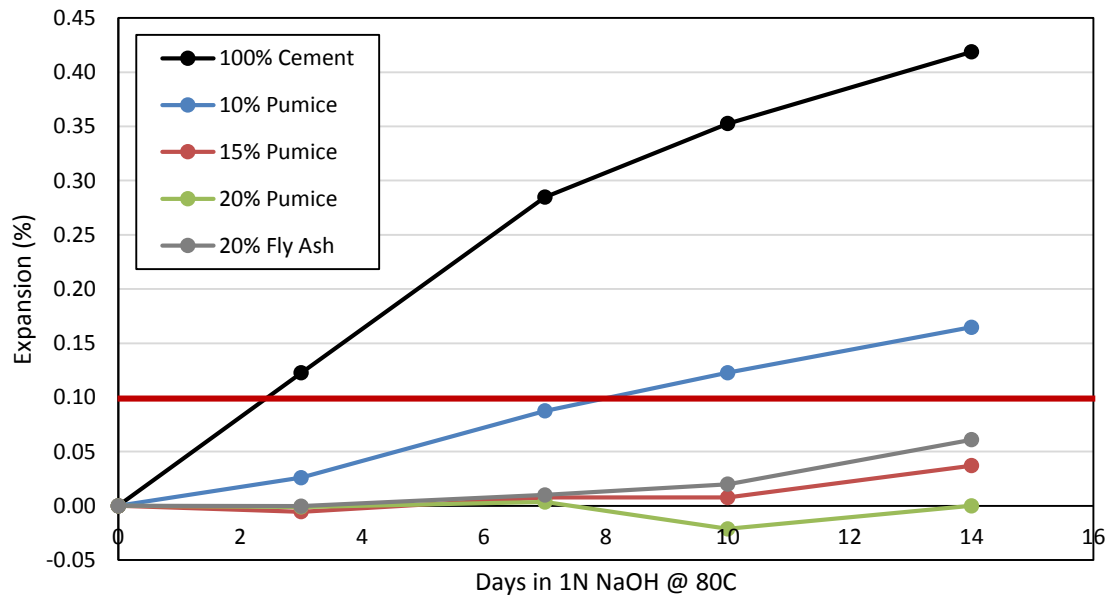


Figure A-1: ASR Mortar Testing Results for Pumice

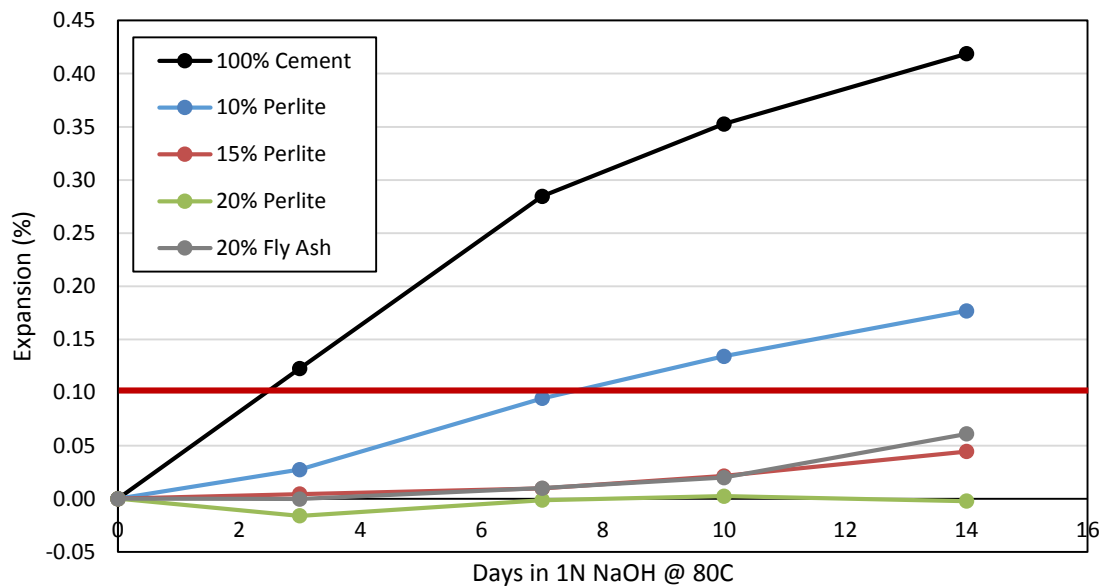


Figure A-2: ASR Mortar Testing Results for Perlite

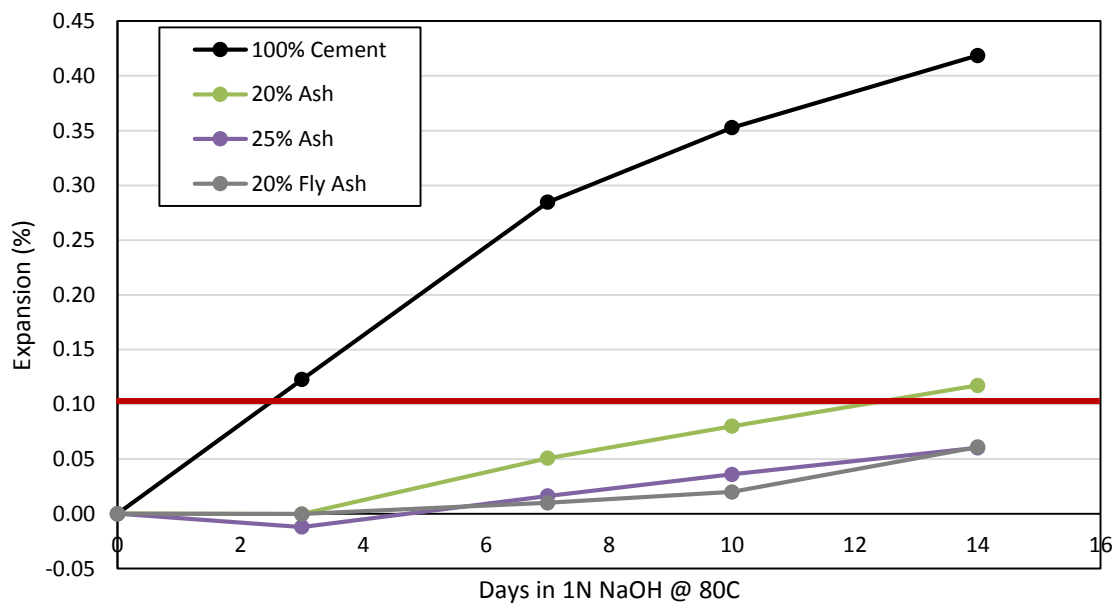


Figure A-3: ASR Mortar Testing Results for Ash

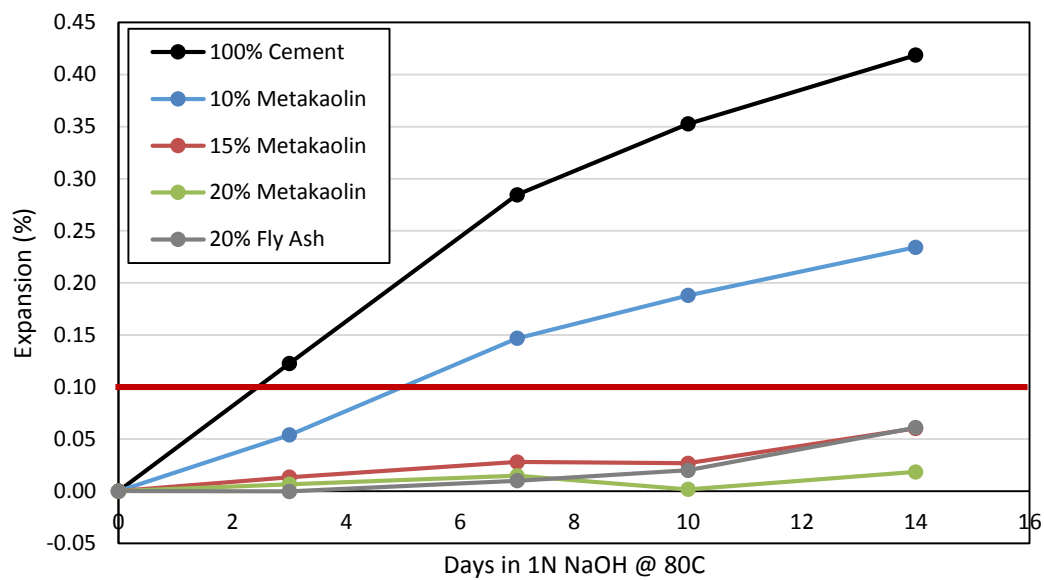


Figure A-4: ASR Mortar Testing Results for Metakaolin

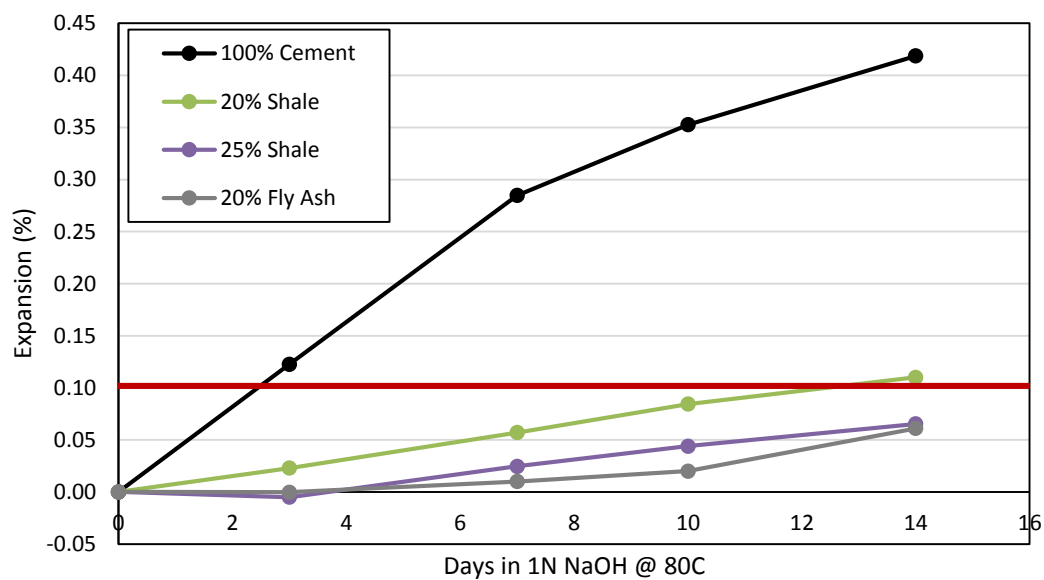


Figure A-5: ASR Mortar Testing Results for Shale

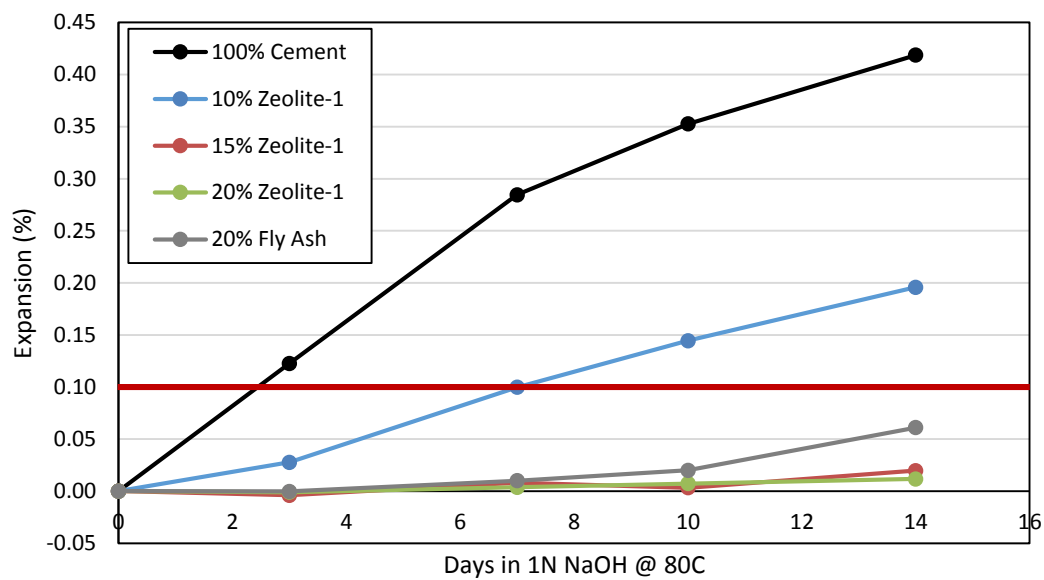


Figure A-6: ASR Mortar Testing for Zeolite-1

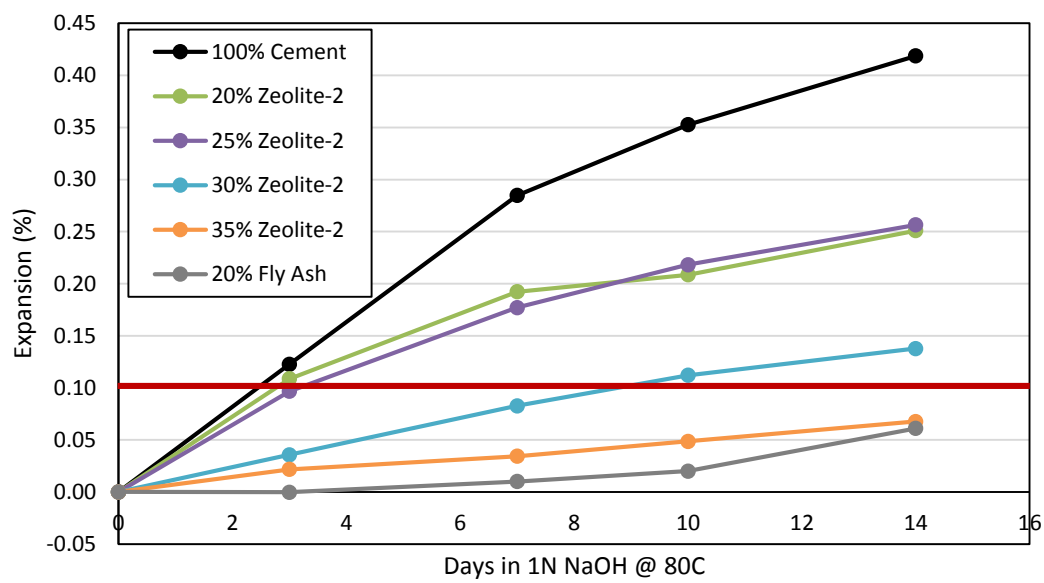


Figure A-7: ASR Mortar Testing Results for Zeolite-2

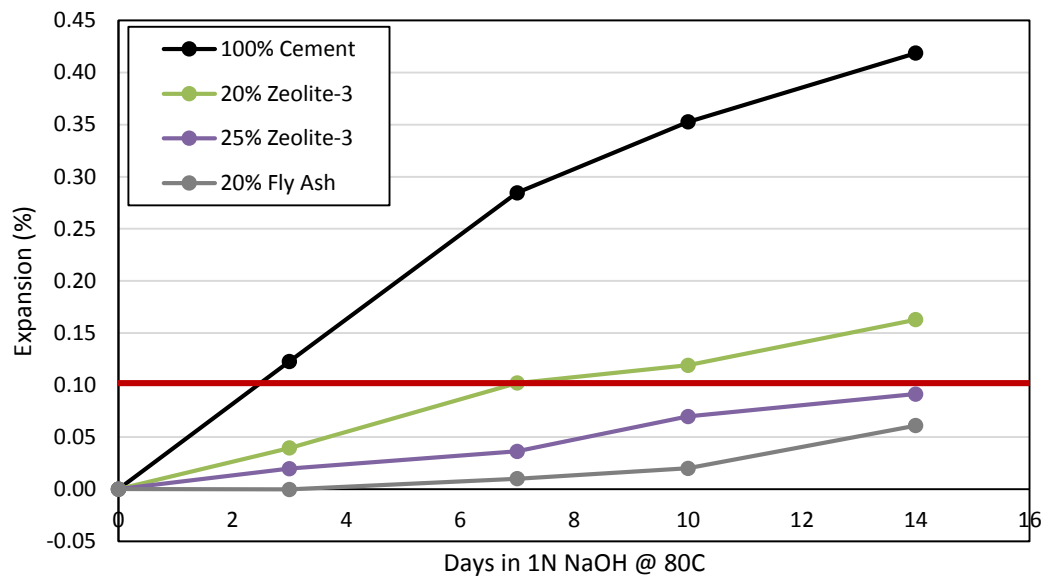


Figure A-8: ASR Mortar Testing Results for Zeolite-3

Appendix B: ASTM C 1293 Results

Table B-1: Concrete Mixture Proportions for Each ASTM C 1293 Mixture
(asterisk indicate the value was assumed)

Description	SCM Density, g/cc	Cement, lb/yd ³	SCM, lb/yd ³	Coarse Agg, lb/yd ³	Fine Agg, lb/yd ³	Water, lb/yd ³	Cement, Vol %	SCM, Vol %	CA, Vol %	FA, Vol %	Water, Vol %	Air ¹⁰ , Vol %
Control	*3.150	564.0	0.0	1937	1257	254	10.6	0.0	43.4	28.9	15.1	2.0
15% Pumice	2.438	479.4	84.6	1937	1257	254	9.0	2.0	43.2	28.8	15.0	2.0
15% Perlite	2.438	479.4	84.6	1937	1257	254	9.0	2.0	43.2	28.8	15.0	2.0
15% Metakaolin	2.748	479.4	84.6	1937	1257	254	9.0	1.8	43.3	28.8	15.0	2.0
15% Zeolite-1	2.363	479.4	84.6	1937	1257	254	9.0	2.1	43.2	28.8	15.0	2.0
15% Fly Ash	*2.500	479.4	84.6	1937	1257	254	9.0	2.0	43.2	28.8	15.0	2.0
25% Pumice	2.438	423.0	141.0	1937	1257	254	7.9	3.4	43.0	28.7	15.0	2.0
25% Perlite	2.438	423.0	141.0	1937	1257	254	7.9	3.4	43.0	28.7	15.0	2.0
25% Ash	2.455	423.0	141.0	1937	1257	254	7.9	3.4	43.1	28.7	15.0	2.0
25% Metakaolin	2.748	423.0	141.0	1937	1257	254	7.9	3.0	43.2	28.8	15.0	2.0
25% Shale	2.583	423.0	141.0	1937	1257	254	7.9	3.2	43.1	28.7	15.0	2.0
25% Zeolite-1	2.363	423.0	141.0	1937	1257	254	7.9	3.5	43.0	28.7	14.9	2.0
35% Zeolite-2	2.460	366.6	197.4	1937	1257	254	6.8	4.7	42.9	28.6	14.9	2.0
25% Zeolite-3	2.290	423.0	141.0	1937	1257	254	7.9	3.6	43.0	28.6	14.9	2.0
25% Fly Ash	*2.500	423.0	141.0	1937	1257	254	7.9	3.3	43.1	28.7	15.0	2.0

¹⁰ Based on preliminary calculations, not actual measurements

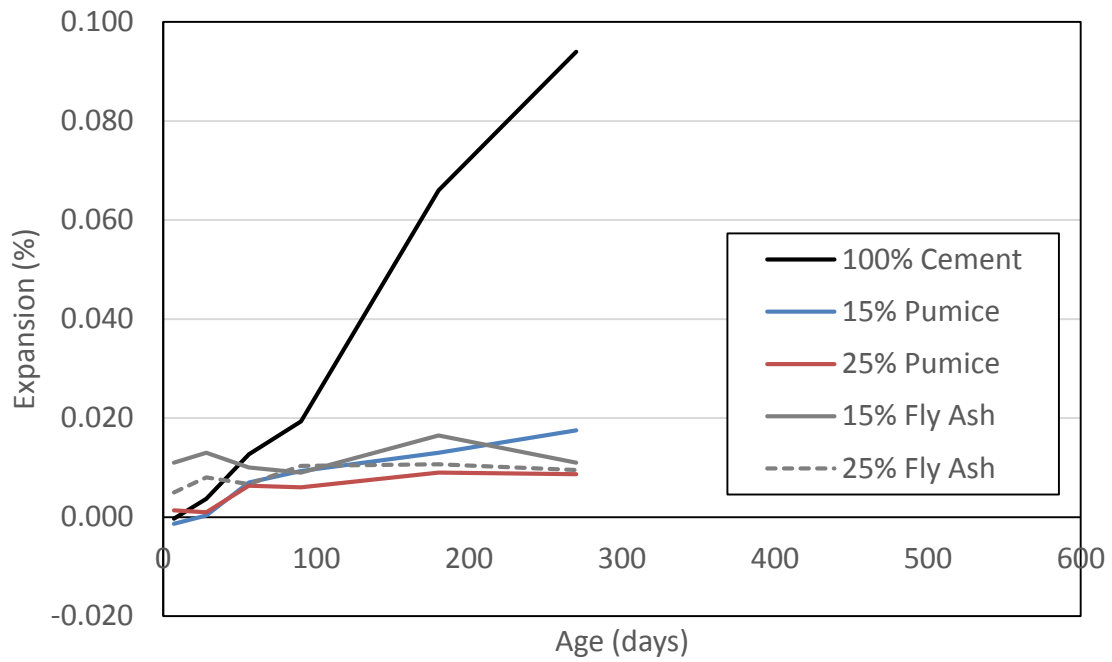


Figure B-1: ASR Concrete Testing Results for Pumice

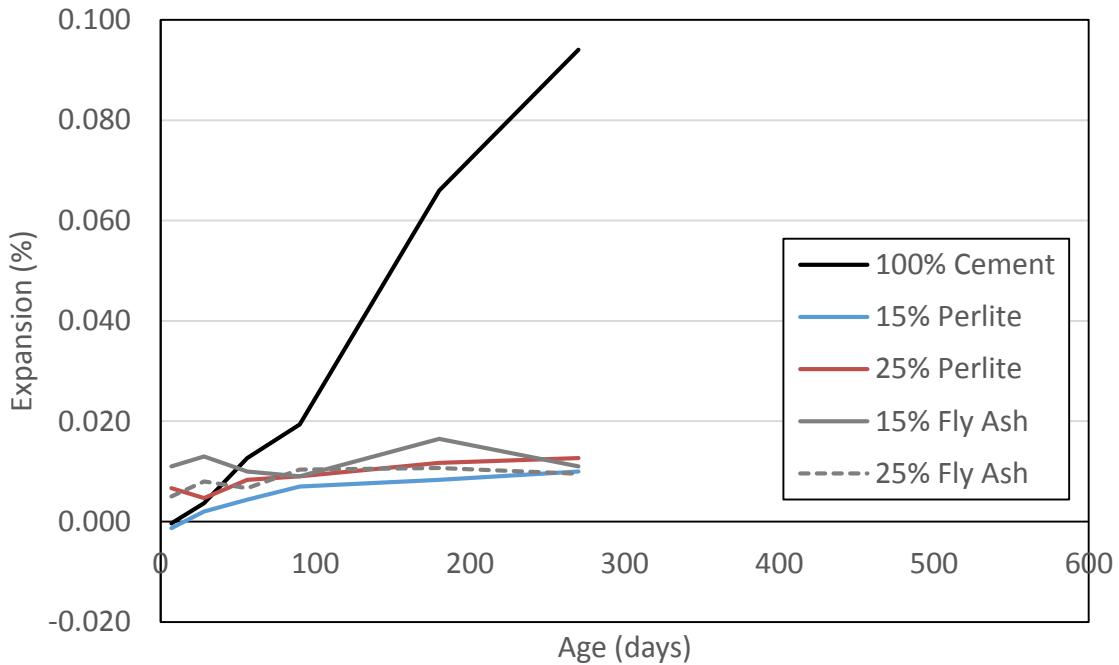


Figure B-2: ASR Concrete Testing Results for Perlite

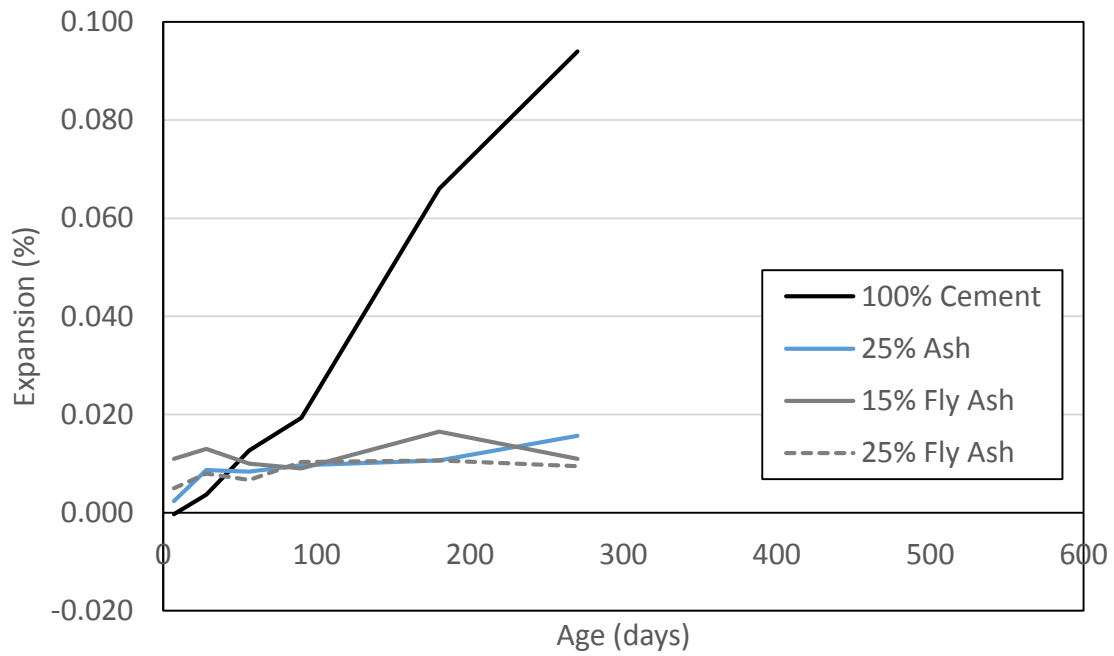


Figure B-3: ASR Concrete Results for Ash

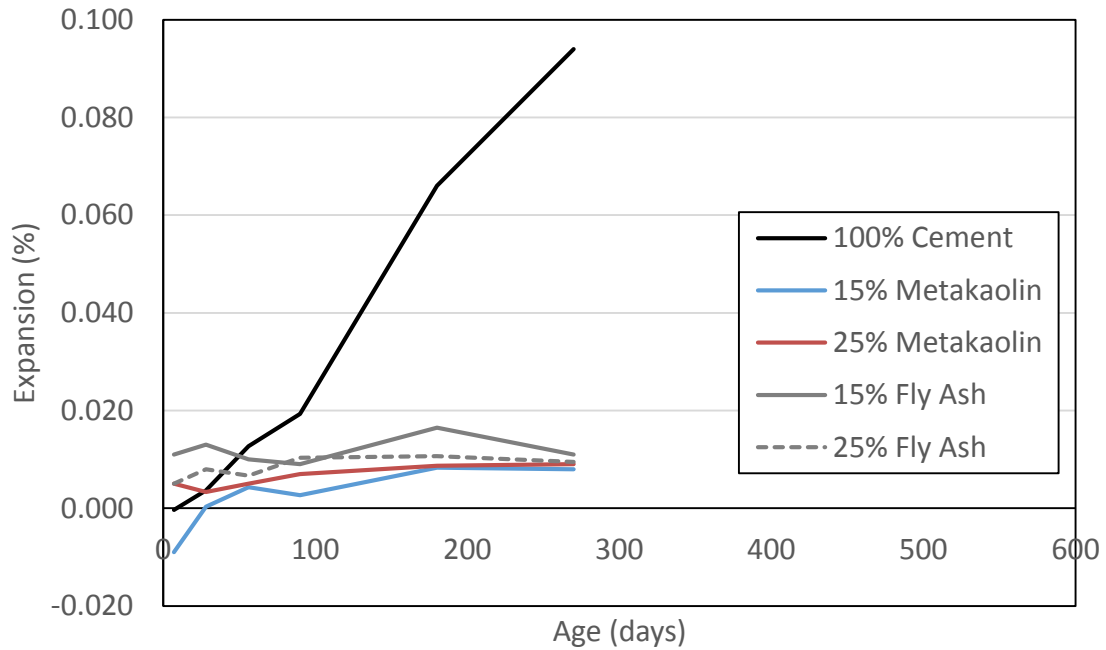


Figure B-4: ASR Concrete Results for Metakaolin

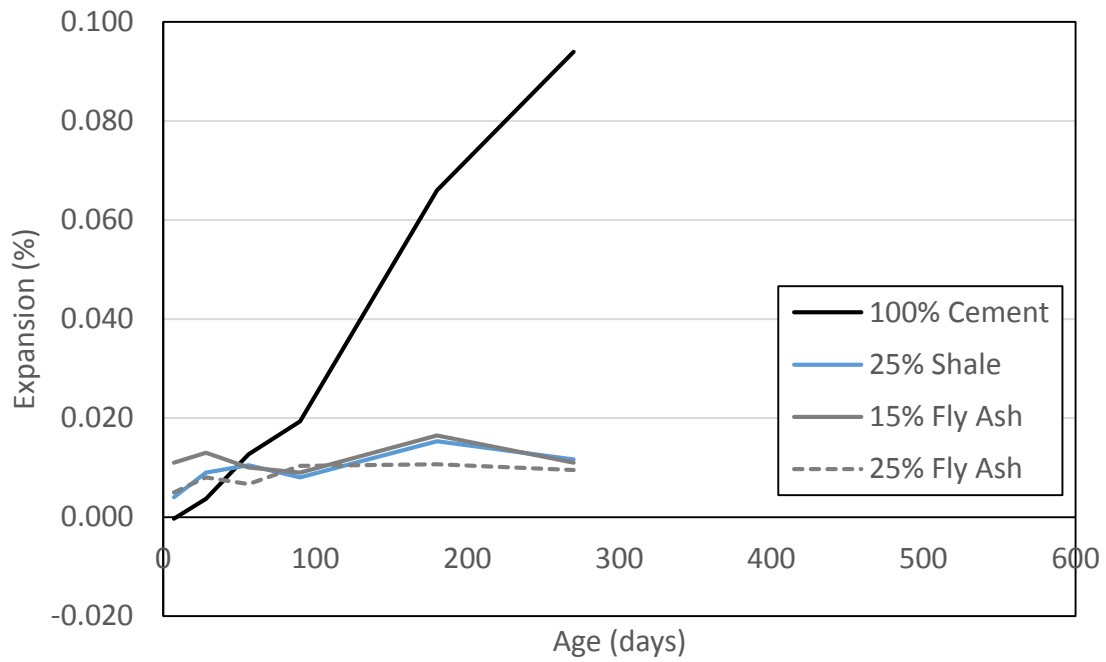


Figure B-5: ASR Concrete Results for Shale

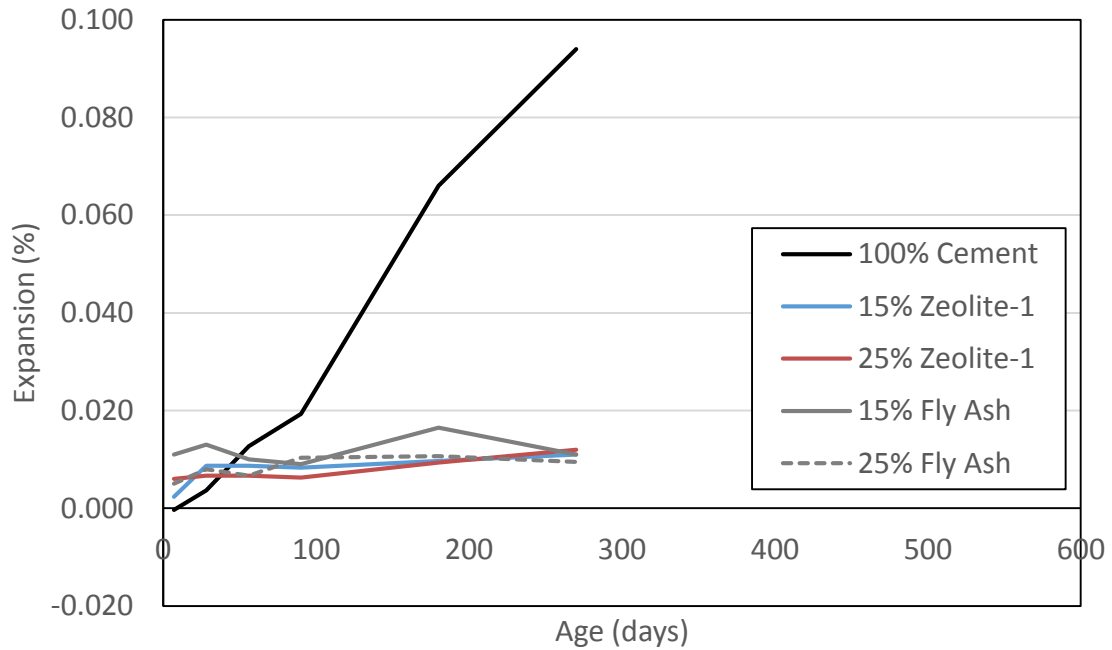


Figure B-6: ASR Concrete Results for Zeolite-1

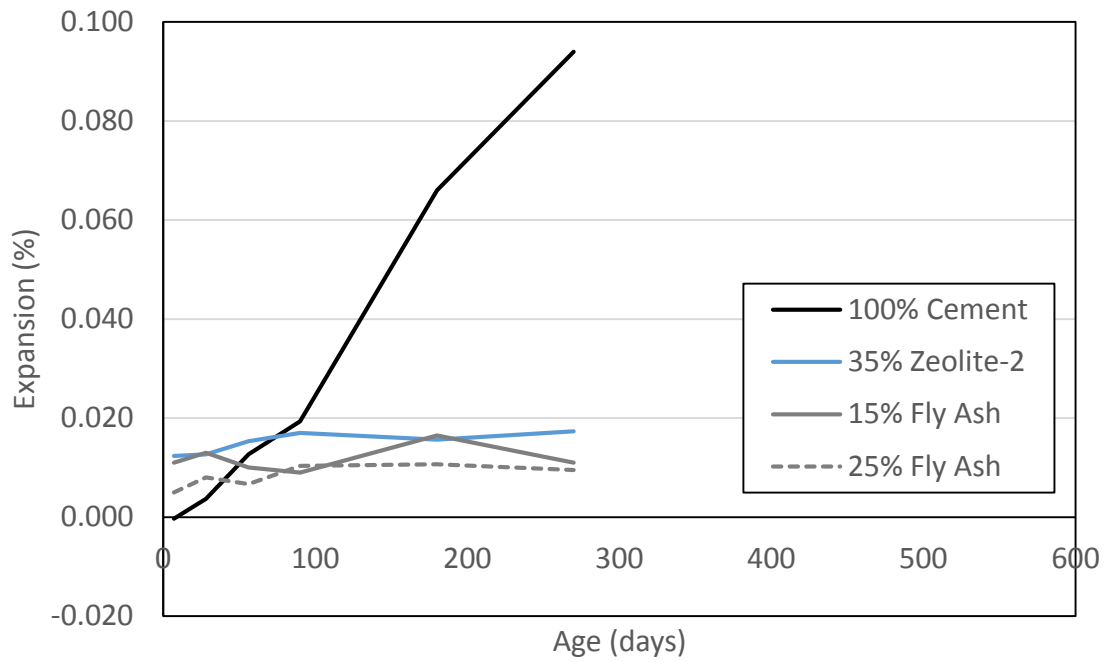


Figure B-7: ASR Concrete Results for Zeolite-2

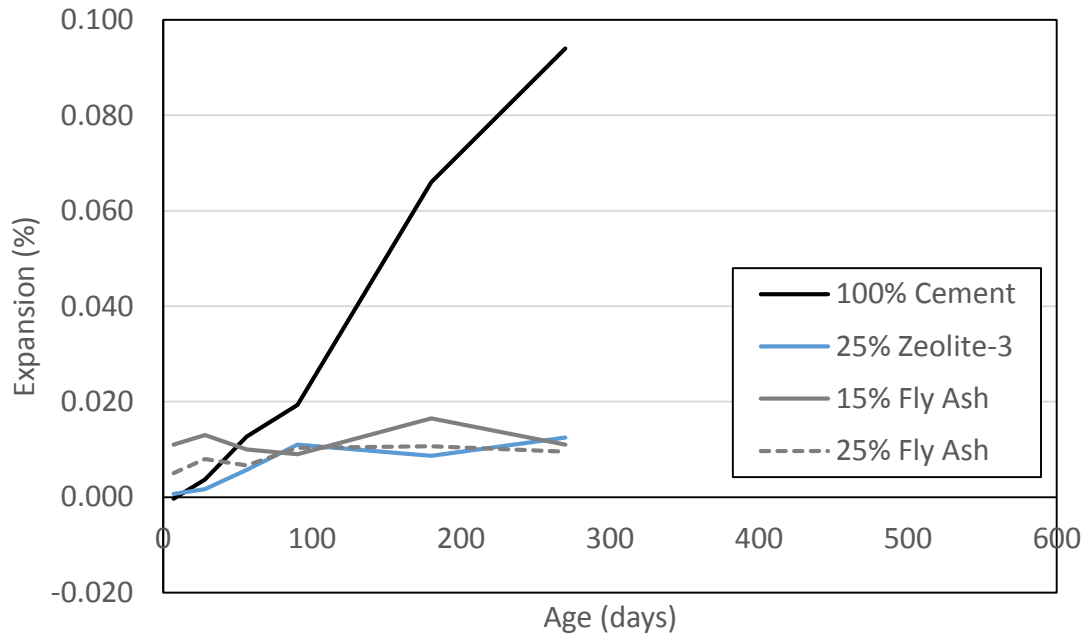


Figure B-8: ASR Concrete Results for Zeolite-3

Appendix C: Fresh State Concrete Properties and Mix Proportions

Table C-1: Measured Fresh Properties of Concrete

Description	Admixture			Slump, in	Air, Vol %	Unit Weight, lb/ft ³
	Pre-dose, g	Post-dose, g	Total, g			
Control	12.0	46.9	58.9	3.25	1.6	150
15% Pumice	12.0	59.8	71.8	2.5	1.8	149.6
25% Pumice	102.1	100.7	202.8	5.25	2.0	148.8
15% Perlite	22.5	22.5	45.0	3	2.2	148.8
25% Perlite	81.4	59.5	140.9	4	2.0	150
25% Ash	100.4	0.0	100.4	4.5	2.0	150.8
15% Metakaolin	20.0	54.8	74.8	3.5	1.8	149.6
25% Metakaolin	80.2	78.1	158.3	3.5	1.8	150
25% Shale	99.2	79.4	178.6	4.75	1.8	147.2
15% Zeolite-1	251.8	95.0	346.8	3	2.4	147.6
25% Zeolite-1	492.6	0.0	492.6	1.5	2.0	148
35% Zeolite-2	190.8	155.7	346.5	3.5	2.1	147.2
25% Zeolite-3	151.0	251.0	402.0	1	-- ¹¹	146.8
15% Fly Ash	0.0	10.4	10.4	3.75	2.0	148.8
25% Fly Ash	0.0	0.0	0.0	5.5	1.6	148.8

¹¹ Air content reading was invalid

Table C-2: Concrete Mixture Proportions for Each Concrete Mixture
(asterisk indicate the value was assumed)

Description	SCM Density, g/cc	Cement, lb/yd ³	SCM, lb/yd ³	Coarse Agg, lb/yd ³	Fine Agg, lb/yd ³	Water, lb/yd ³	Cement, Vol %	SCM, Vol %	CA, Vol %	FA, Vol %	Water, Vol %	Air, Vol %
Control	*3.150	564	0	1937	1277	254	10.6	0.0	43.4	29.4	15.1	2.0
15% Pumice	2.438	479.4	84.6	1937	1277	254	8.9	2.0	43.0	29.1	14.9	2.0
15% Perlite	2.438	479.4	84.6	1937	1277	254	8.9	2.0	43.0	29.1	14.9	2.0
15% Metakaolin	2.748	479.4	84.6	1937	1277	254	9.0	1.8	43.1	29.2	15.0	2.0
15% Zeolite-1	2.363	479.4	84.6	1937	1277	254	8.9	2.1	43.0	29.1	14.9	2.0
15% Fly Ash	*2.500	479.4	84.6	1937	1277	254	9.0	2.0	43.0	29.1	14.9	2.0
25% Pumice	2.438	423	141	1937	1277	254	7.9	3.4	42.9	29.0	14.9	2.0
25% Perlite	2.438	423	141	1937	1277	254	7.9	3.4	42.9	29.0	14.9	2.0
25% Ash	2.455	423	141	1937	1277	254	7.9	3.4	42.9	29.0	14.9	2.0
25% Metakaolin	2.748	423	141	1937	1277	254	7.9	3.0	43.0	29.1	14.9	2.0
25% Shale	2.583	423	141	1937	1277	254	7.9	3.2	42.9	29.1	14.9	2.0
25% Zeolite-1	2.363	423	141	1937	1277	254	7.9	3.5	42.8	29.0	14.9	2.0
35% Zeolite-2	2.460	366.6	197.4	1937	1277	254	6.8	4.7	42.7	28.9	14.9	2.0
25% Zeolite-3	2.290	423	141	1937	1277	254	7.9	3.6	42.8	29.0	14.9	2.0
25% Fly Ash	*2.500	423	141	1937	1277	254	7.9	3.3	42.9	29.0	14.9	2.0

Appendix D: Hardened Concrete Testing Results

Table D-1: Concrete Compressive Strength Testing
(red values indicate the measured strength fell outside the acceptable range of values and was not counted towards the averages shown in Table 3-8)

Concrete Description	7 days			28 days			56 days			90 days		
	Cylinder 1	Cylinder 2	Cylinder 3	Cylinder 1	Cylinder 2	Cylinder 3	Cylinder 1	Cylinder 2	Cylinder 3	Cylinder 1	Cylinder 2	Cylinder 3
Control	5703	5555	5677	6427	6759	7051	7064	7006	7157	7553	7568	7136
15% Pumice	5134	5341	4968	5956	6100	6216	6427	6641	6751	6924	7129	7097
15% Perlite	4230	4097	4193	5296	5403	4975	5656	5876	5953	6065	6149	6489
15% Metakaolin	5377	5193	5076	6713	6809	6708	6942	7284	7508	7334	7617	7398
15% Zeolite-1	6139	6348	5389	7626	8092	8213	8574	8180	8150	8201	8044	8516
15% Fly Ash	5034	5428	5090	6427	7101	6461	7640	8118	7785	8337	7780	8183
25% Pumice	4554	4432	4631	6120	6198	6068	6934	6743	6450	7402	7551	7120
25% Perlite	4076	4295	4083	5105	5415	5784	6505	6413	5889	7152	6544	6451
25% Ash	3874	4100	3949	5591	5192	4978	5394	5698	5913	6302	6330	6568
25% Metakaolin	6381	6300	6359	7373	7439	7635	429	453	471	7817	7842	7983
25% Shale	4800	4940	4886	6516	6451	6542	7331	7147	7618	7649	7441	7454
25% Zeolite-1	5329	4748	5619	7497	7228	7511	7445	7257	6829	7656	7432	7028
35% Zeolite-2	3454	3146	3225	4847	4975	4749	5523	5584	5457	5319	5457	5766
25% Zeolite-3	4163	4387	4296	5885	5580	5914	6396	6466	6299	6000	6653	6000
25% Fly Ash	4510	4692	4569	6378	6236	5769	7568	7173	7053	7776	7450	7817

Table D-2: ASTM C 596 Drying Shrinkage Results¹²

Mortar Description	4 day		11 day		18 day		25 day		56 day		112 day	
	DS ¹³ , %	WL ¹⁴ , %	DS, %	WL, %	DS, %	WL, %	DS, %	WL, %	DS, %	WL, %	DS, %	WL, %
Control	0.06	2.31	0.08	2.97	0.09	3.29	0.11	3.49	0.12	3.82	0.12	3.73
20% Pumice	0.06	3.76	0.09	4.25	0.11	4.68	0.12	4.83	0.13	4.97	0.14	4.93
20% Perlite	0.06	3.51	0.09	4.25	0.10	4.49	0.10	4.61	0.12	4.71	0.13	4.70
20% Ash	0.07	3.44	0.10	4.19	0.11	4.45	0.12	4.58	0.12	4.77	0.13	4.78
20% Metakaolin	0.07	3.63	0.10	4.18	0.11	4.36	0.11	4.45	0.13	4.55	0.14	4.54
20% Shale	0.06	3.87	0.08	4.63	0.10	4.92	0.10	5.03	0.11	5.23	0.12	5.20
20% Zeolite-1	0.08	6.56	0.11	7.17	0.12	7.29	0.12	7.34	0.13	7.25	0.15	6.83
20% Zeolite-2	0.08	5.69	0.12	6.59	0.13	6.83	0.14	6.92	0.15	6.87	0.16	6.52
20% Zeolite-3	0.10	7.07	0.14	7.85	0.16	8.02	0.16	8.05	0.18	7.94	0.20	7.52
20% Fly Ash	0.05	3.02	0.07	3.44	0.09	3.84	0.10	3.99	0.11	4.26	0.12	4.30

¹² All results are the averages of 3 or 4 mortar bars

¹³ DS = Drying Shrinkage

¹⁴ WL = Weight Loss

Table D-3: ASTM C 157 Drying Shrinkage Results¹⁵

Concrete Description	4 day		7 day		14 day		28 day		56 day		112 day		224 day	
	DS ¹⁶ , %	WL ¹⁷ , %	DS, %	WL, %	DS, %	WL, %	DS, %	WL, %	DS, %	WL, %	DS, %	WL, %	DS, %	WL, %
Control	0.004	0.955	0.011	1.112	0.015	1.303	0.017	1.505	0.021	1.733	0.028	1.957	0.027	2.168
15% Pumice	0.016	1.108	0.014	1.268	0.019	1.505	0.028	1.734	0.032	1.981	0.039	2.227	0.041	2.444
25% Pumice	0.012	1.057	0.015	1.247	0.021	1.484	0.037	1.713	0.044	1.987	0.045	2.237	0.046	2.396
15% Perlite	0.018	1.161	0.016	1.338	0.023	1.562	0.027	1.801	0.033	2.051	0.040	2.295	0.041	2.496
25% Perlite	0.015	1.177	0.017	1.370	0.030	1.608	0.033	1.865	0.041	2.122	0.048	2.359	0.000	0.000
25% Ash	0.014	1.303	0.018	1.532	0.025	1.812	0.032	2.105	0.038	2.404	0.046	2.686	0.050	2.854
15% Metakaolin	0.006	0.760	0.009	0.887	0.012	1.069	0.019	1.278	0.027	1.479	0.031	1.704	0.036	1.909
25% Metakaolin	0.008	0.783	0.010	0.944	0.019	1.141	0.023	1.366	0.031	1.642	0.035	1.842	0.000	0.000
25% Shale	0.000	0.000	0.013	1.349	0.020	1.602	0.028	1.858	0.034	2.134	0.038	2.367	0.000	0.000
15% Zeolite-1	0.006	0.761	0.008	0.908	0.013	1.108	0.023	1.327	0.026	1.561	0.031	1.813	0.036	2.021
25% Zeolite-1	0.003	0.800	0.003	0.950	0.009	1.178	0.022	1.451	0.026	1.736	0.031	1.994	0.000	0.000
35% Zeolite-2	0.012	1.401	0.017	1.610	0.023	1.966	0.033	2.307	0.043	2.653	0.046	2.959	0.000	0.000
25% Zeolite-3	0.010	1.126	0.014	1.338	0.023	1.641	0.036	1.940	0.044	2.301	0.052	2.653	0.057	2.865
15% Fly Ash	0.013	1.039	0.014	1.227	0.021	1.467	0.025	1.736	0.028	2.006	0.037	2.248	0.000	0.000
25% Fly Ash	0.005	1.129	0.015	1.327	0.016	1.587	0.019	1.869	0.027	2.145	0.028	2.373	0.000	0.000

¹⁵ All results are the averages of 3 or 4 concrete prisms¹⁶ DS = Drying Shrinkage¹⁷ WL = Weight Loss

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